Gold-catalyzed tandem reaction in water: An efficient and convenient synthesis of fused polycyclic indoles

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Contents

The Crystal Structure of 3i by X-ray Analysis.

X-ray crystallographic data of **3i** were solutions at T = 132(2) K: $C_{15}H_{15}ClN_2O$, M_r = 274.74, Monoclinic, Space group *p2(1)*, a = 7.062(3) Å, b = 7.575(4) Å, c = 12.479(4) Å, α=90°, β=102.770(7)°, γ= 90°, *V*=651.1(5) $Å^3$, *Z*=2.. CCDC 857524 contains the supplementary crystallographic data for this paper.

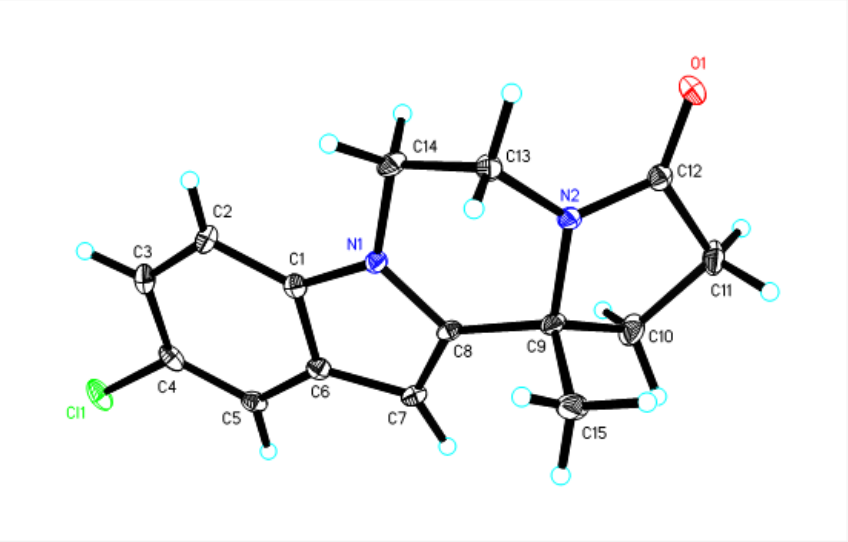


Figure 1. The Crystal Structure of **3i** by X-ray Analysis.

These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

General Information

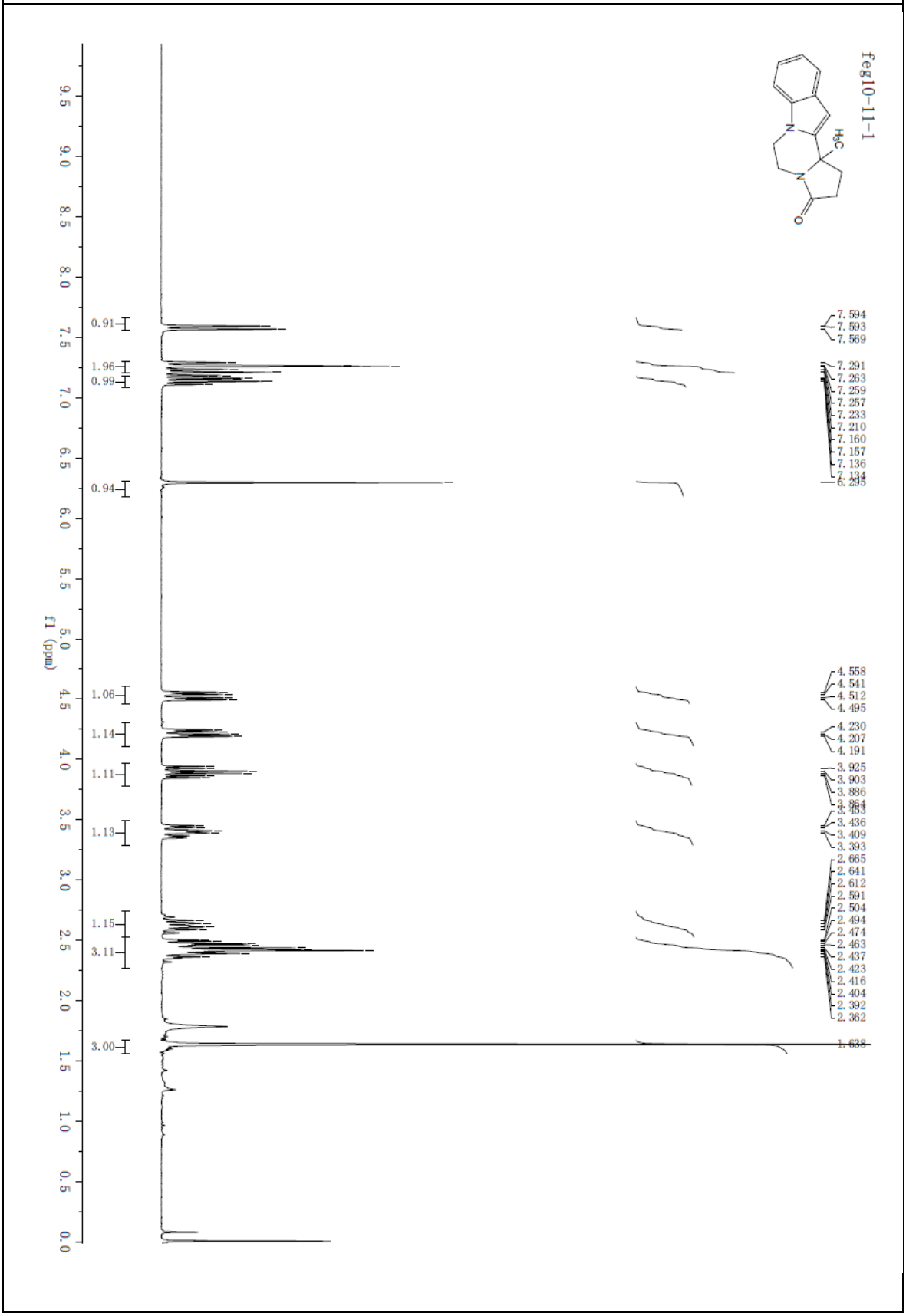
Microwave method in one-pot process for compound **3a-3h**, **3Aa**, **3Ab**, **3Ah** and **3Ai**. A mixture of 2-(1*H*-indol-1-yl) ethanamines 1 (0.2 mmol), 4-pentynoic acid 2 (0.3 mmol) and Au catalyst X (0.02 mmol) was stirred in water (2 mL) under air. The vial was sealed and the mixture was then irradiated for 30 min at 150 ^{o}C. After the reaction was cooled to ambient temperature, the crude reaction mixture was extracted three times with ethyl acetate (EA) (3 x 15 mL). The combined organic phase was washed with saturated $NaHCO_3$ solution, brine, dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography on Combi*Flash*® to provide the desired product.

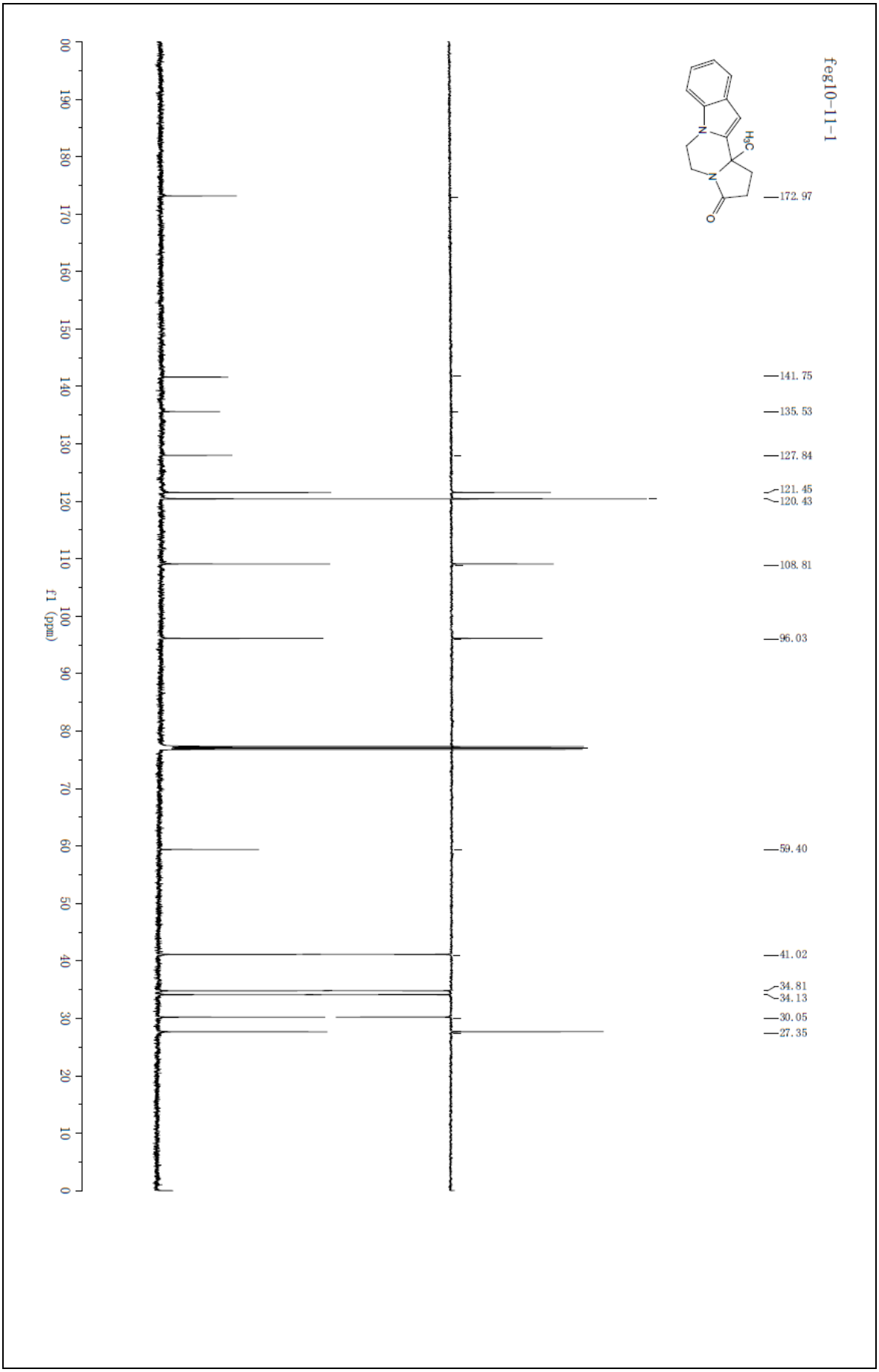
Microwave method in the two-step one-pot process for compound 3h-3s, 3Aa-3Al. A mixture of 2-(1*H*-indol-1-yl) ethanamines 1 (0.2 mmol), alkynoic acid 2 (0.3 mmol) and Au catalyst X (0.02 mmol) was stirred in water (2 mL) under air. The vial was sealed and the mixture was then irradiated for 30 min at 150 ^{o}C. After the reaction was cooled, CF_3COOH (0.2 mmol) was added into the reaction mixture, then irradiated for another 30 min at 150 ^{o}C. After the reaction was cooled to ambient temperature, the crude reaction mixture was extracted three times with ethyl acetate (EA) (3 x 15 mL). The combined organic phase was washed with saturated $NaHCO_3$ solution, brine, dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography on Combi*Flash*® to provide the desired product.

Classical method using a thermostatted oil bath for compound 3a, 3b and 3e. A mixture of 2-(1*H*-indol-1-yl) ethanamines 1 (0.2 mmol), 4-pentynoic acid 2 (0.3 mmol) and Au catalyst (0.02 mmol) was stirred in water (2 mL) under air. The vial was sealed and the mixture was then stirred at 100 ^{o}C with oil heating for 16 h. After the reaction was cooled to ambient temperature, the crude reaction mixture was extracted three times with ethyl acetate (EA) (3 x 15 mL). The combined organic phase was washed with saturated $NaHCO_3$ solution, brine, dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography on Combi*Flash*® to provide the desired product.

Copies of ^{1}H NMR and ^{13}C NMR of Fused Polycyclic Indoles

Compound 3a





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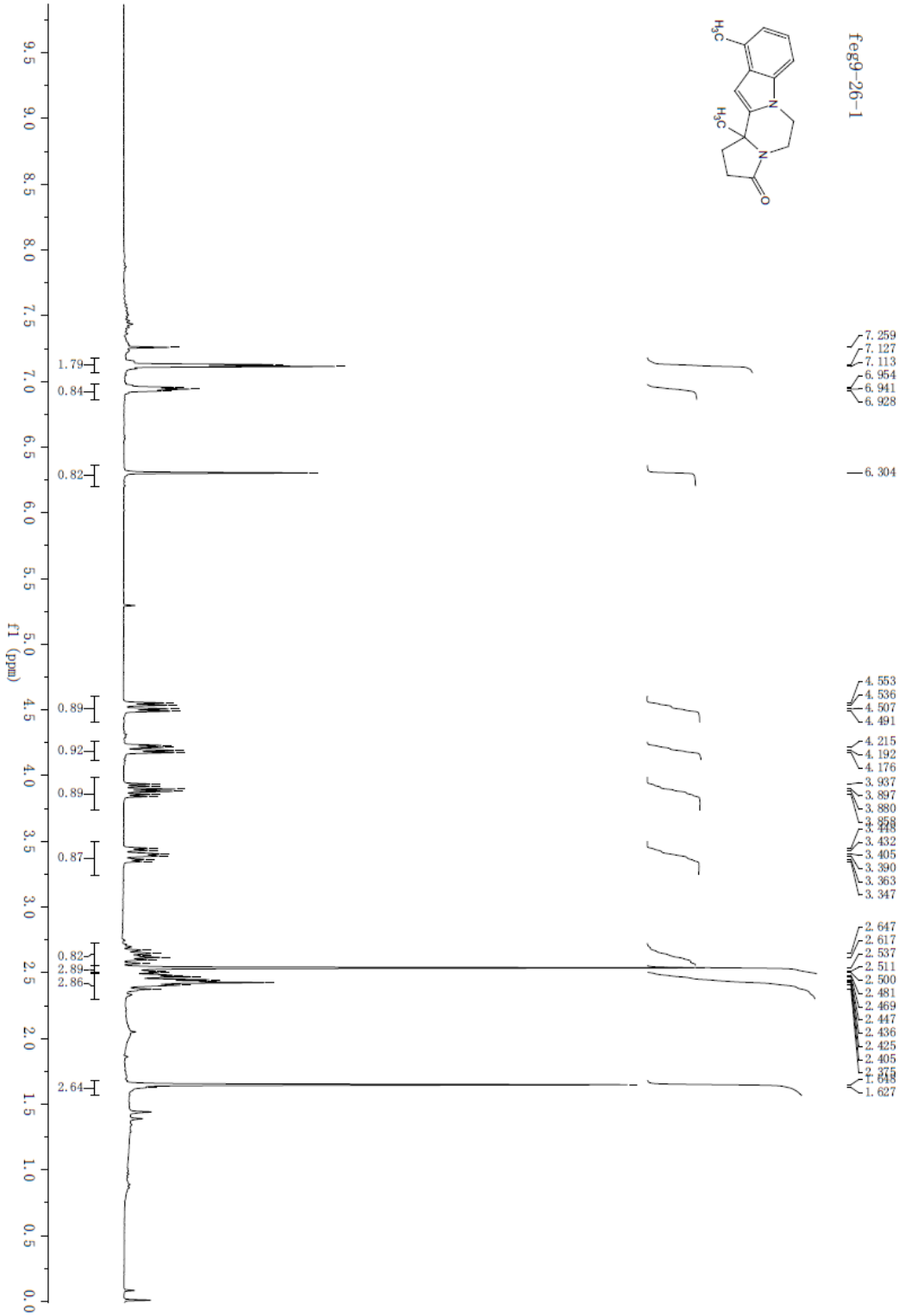
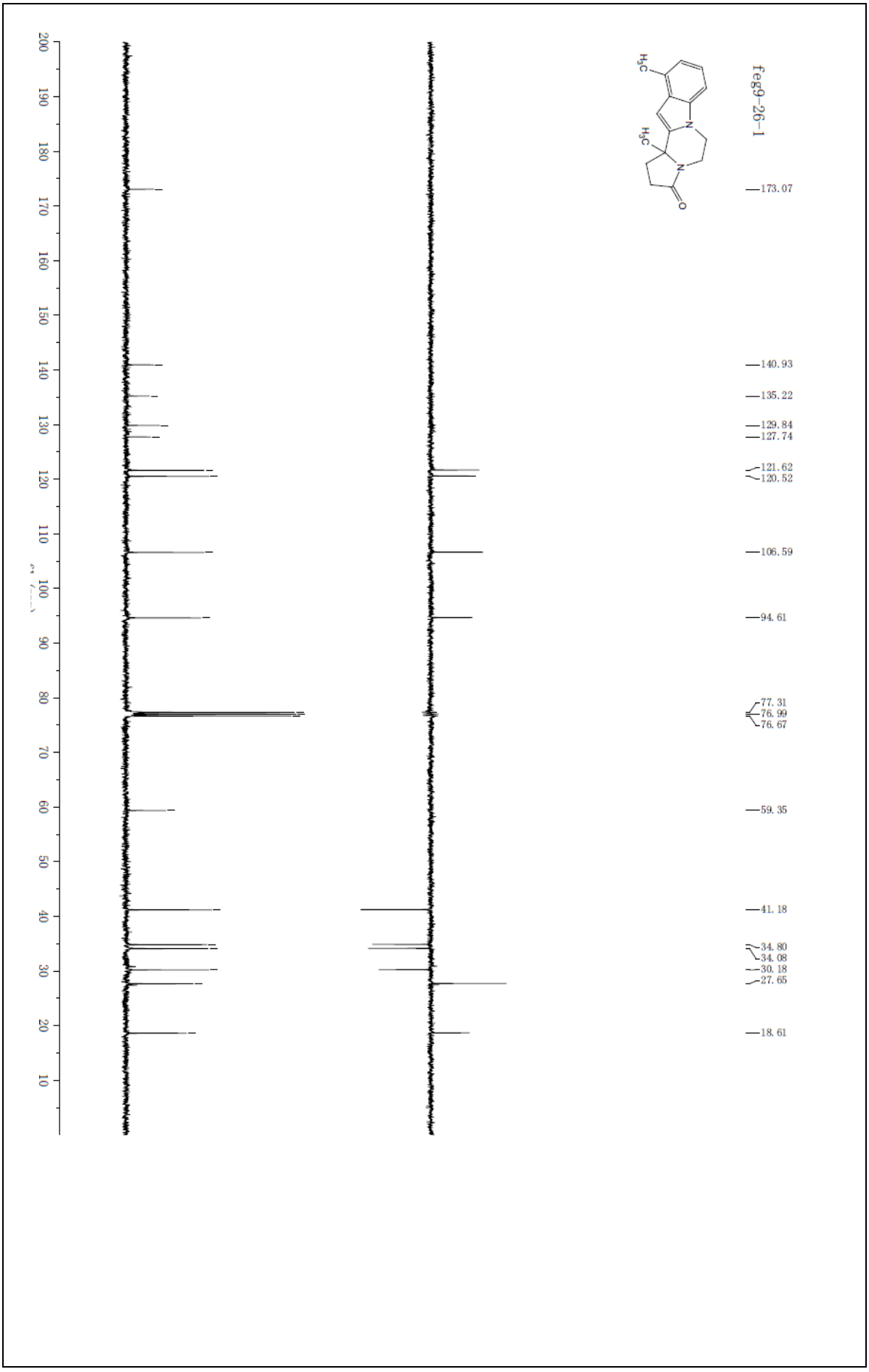
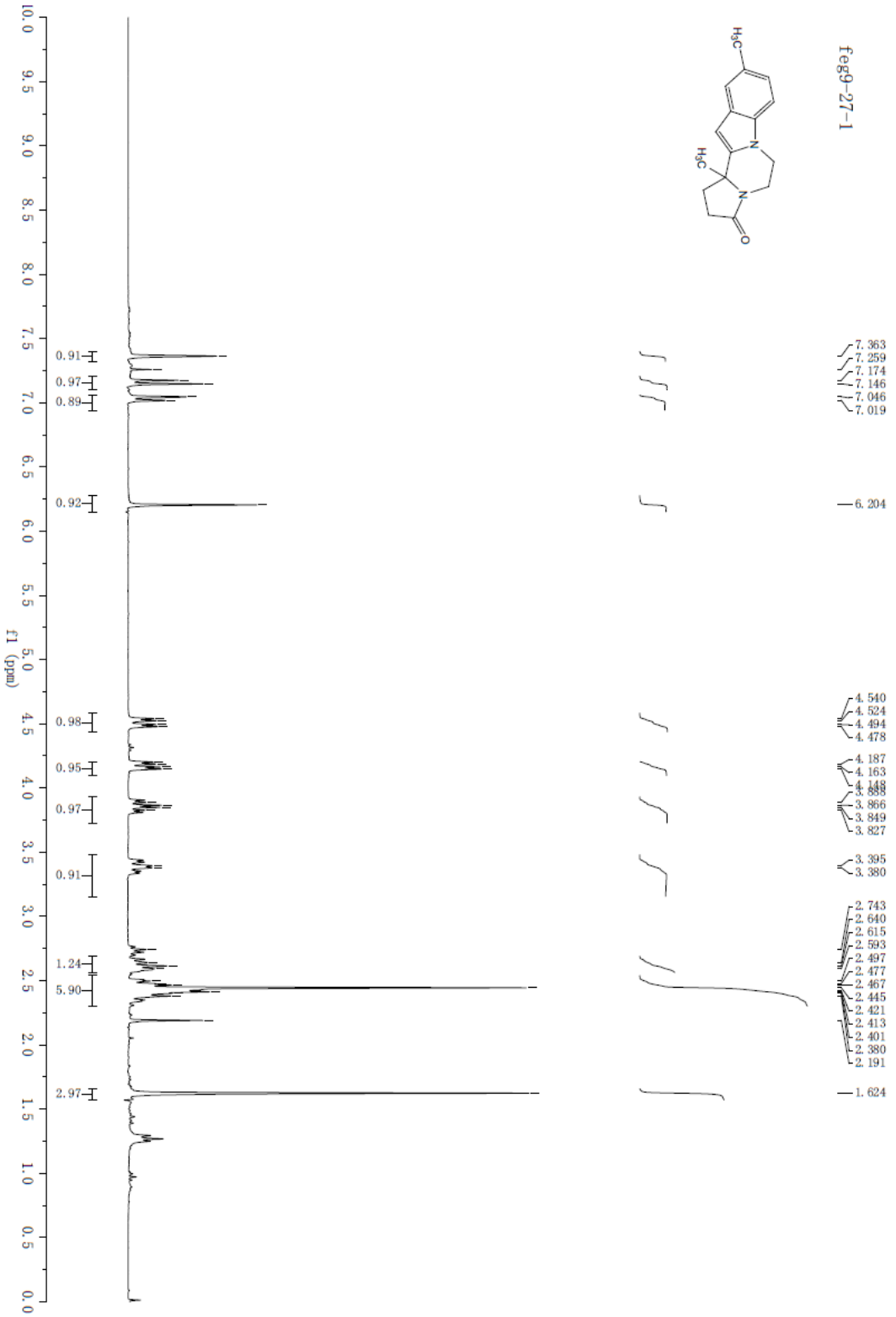
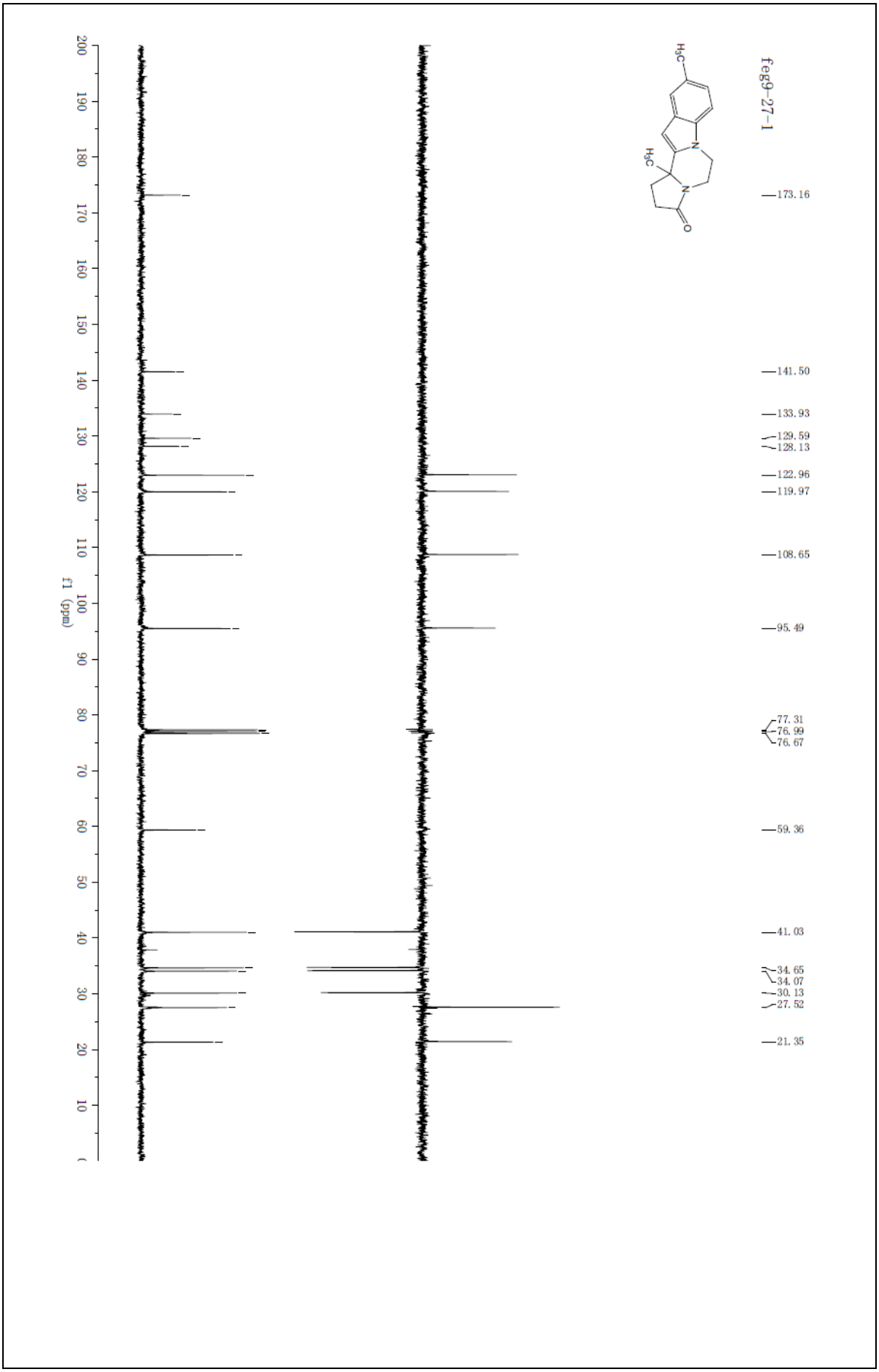


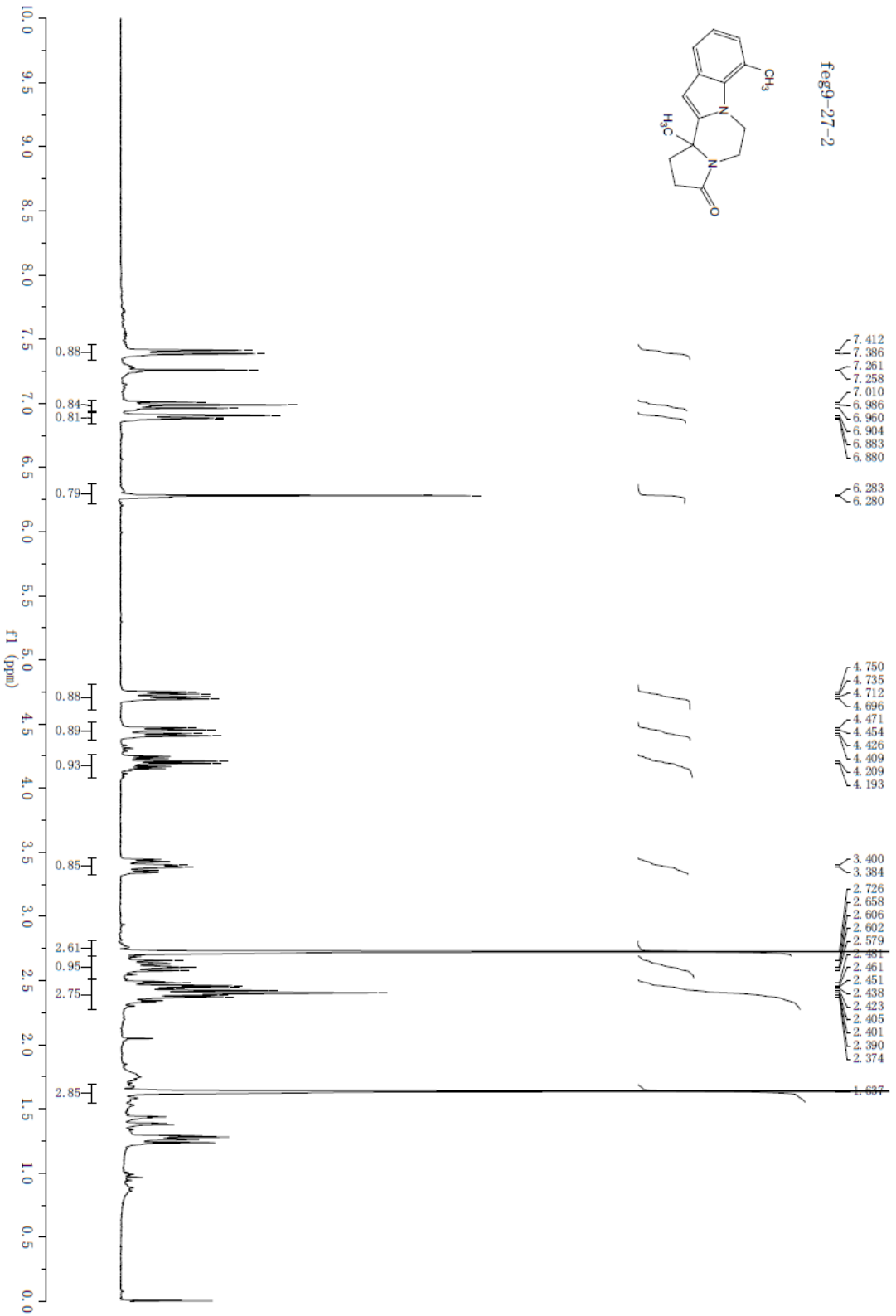


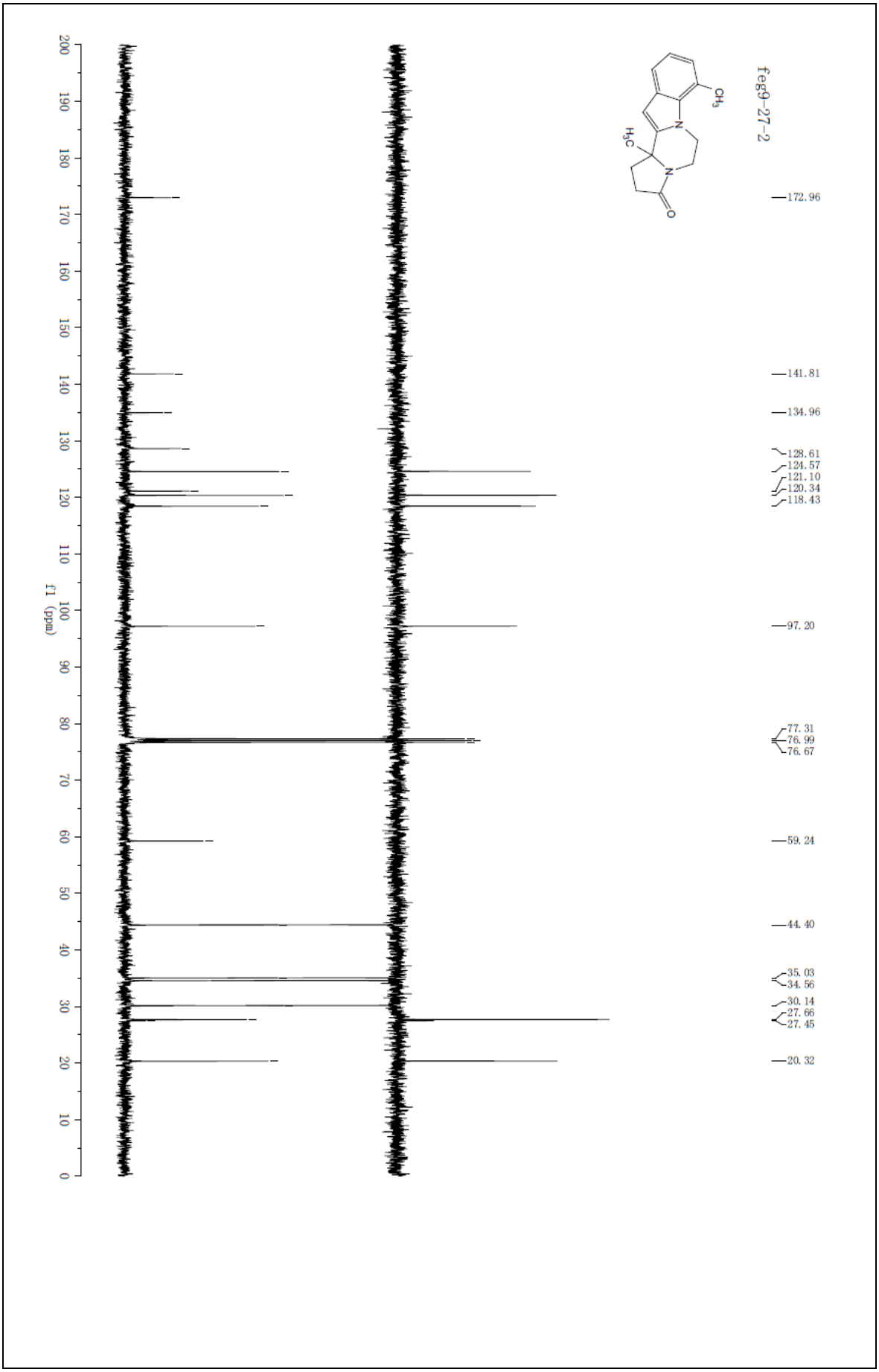
Compound 3c





Compound 3d





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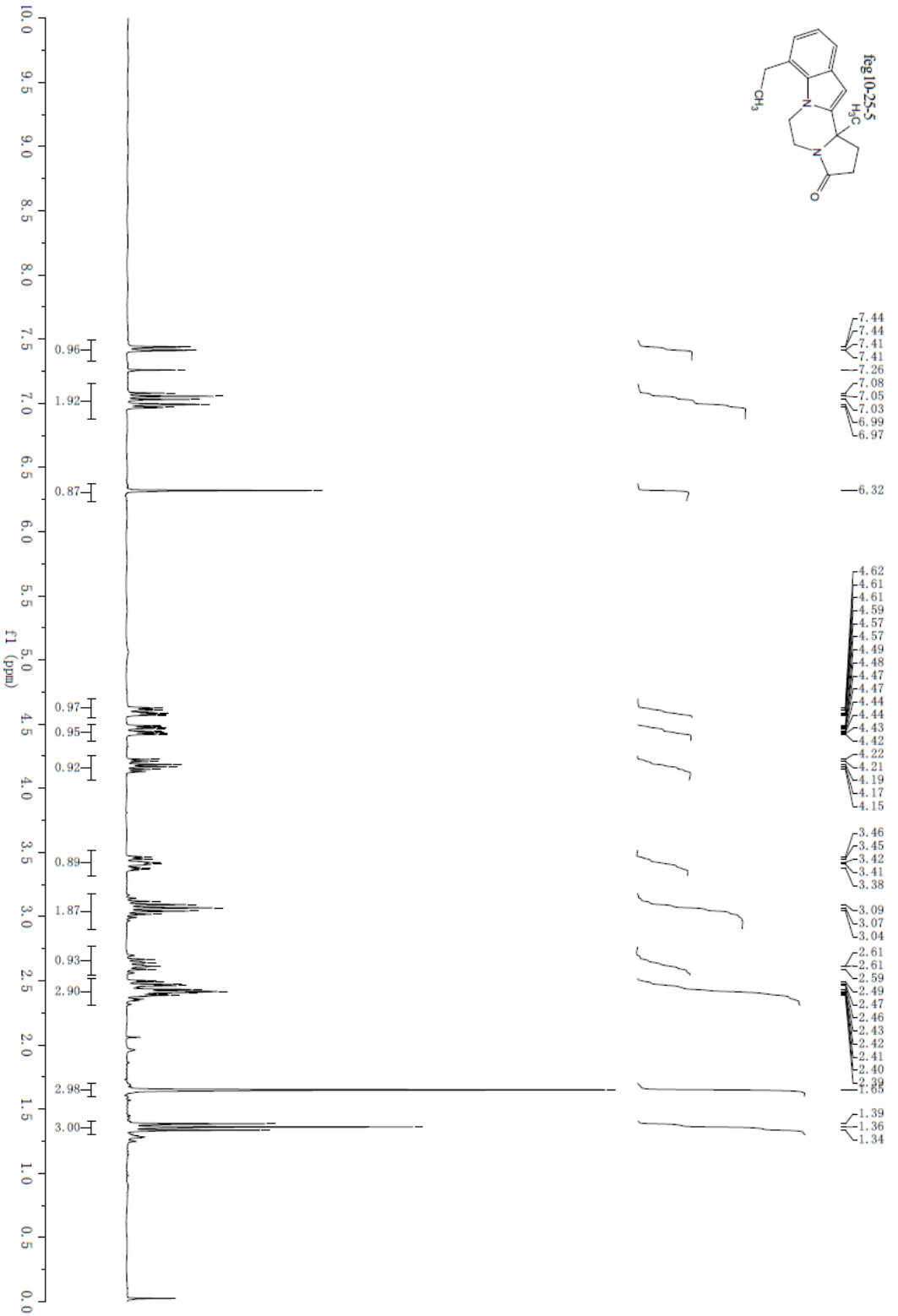

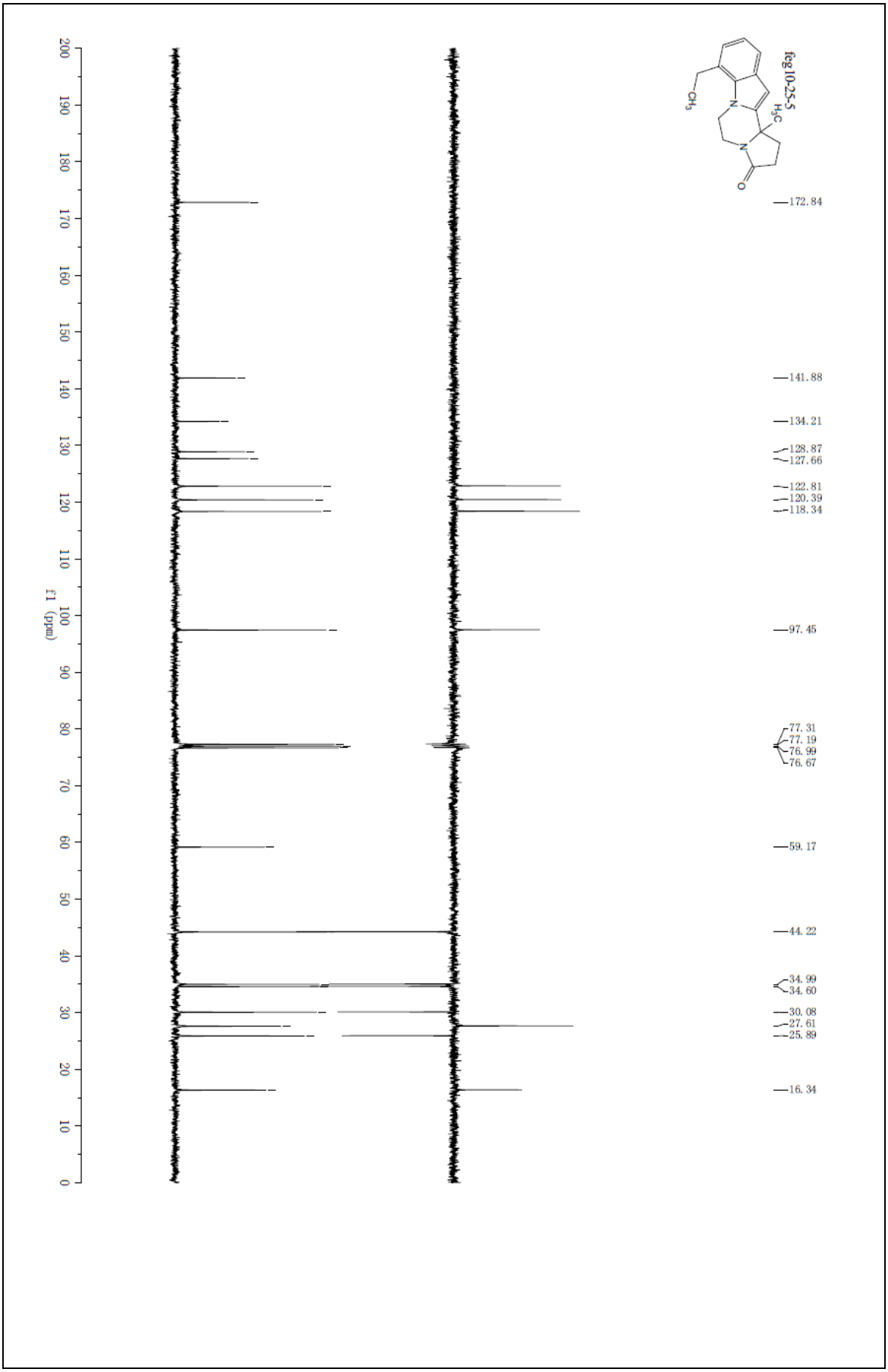
feg10-25-5
CH3
H3C
N
N
O
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
172.84
141.88
134.21
128.87
127.66
122.81
120.39
118.34
97.45
77.31
77.19
76.99
76.67
59.17
44.22
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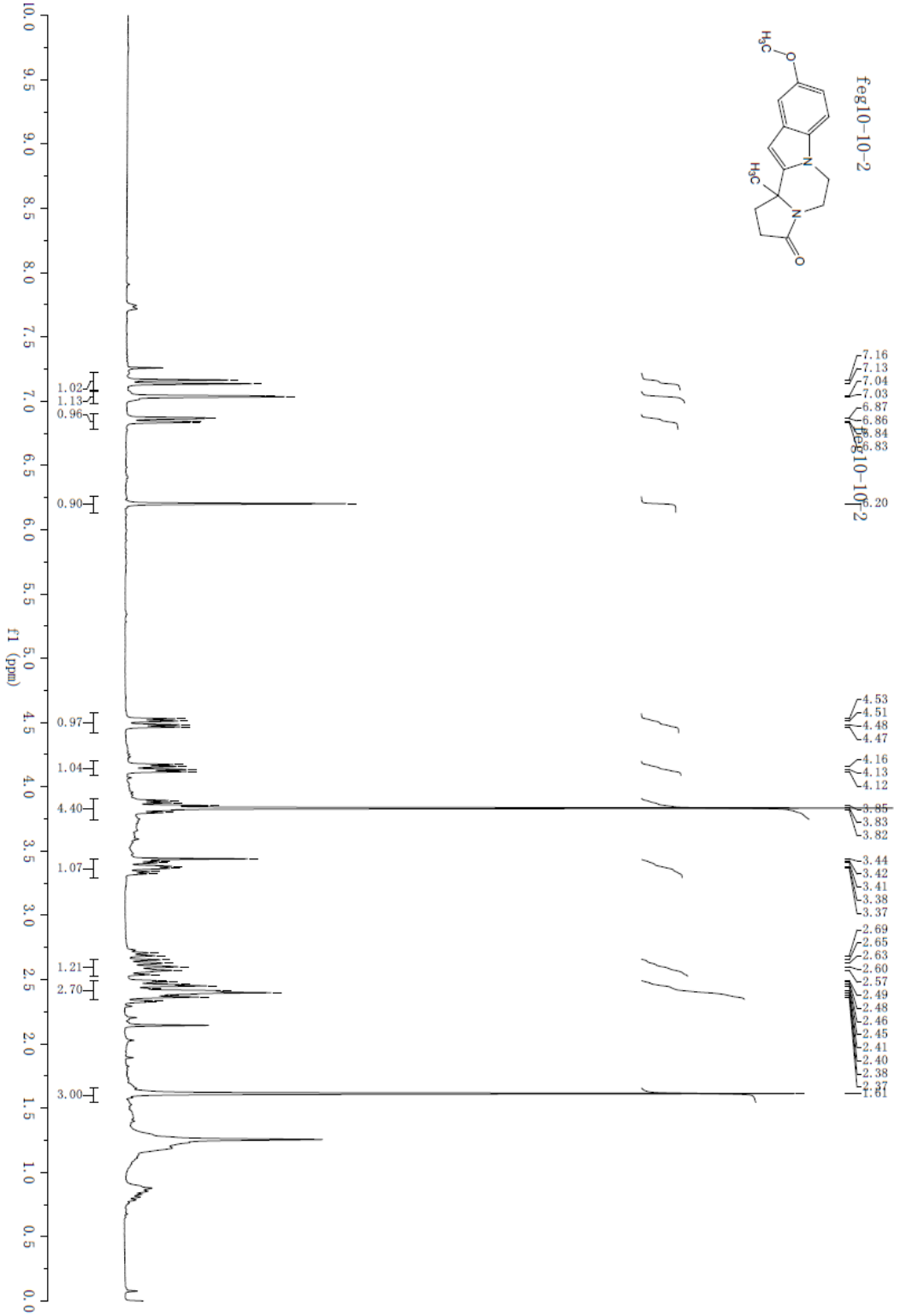
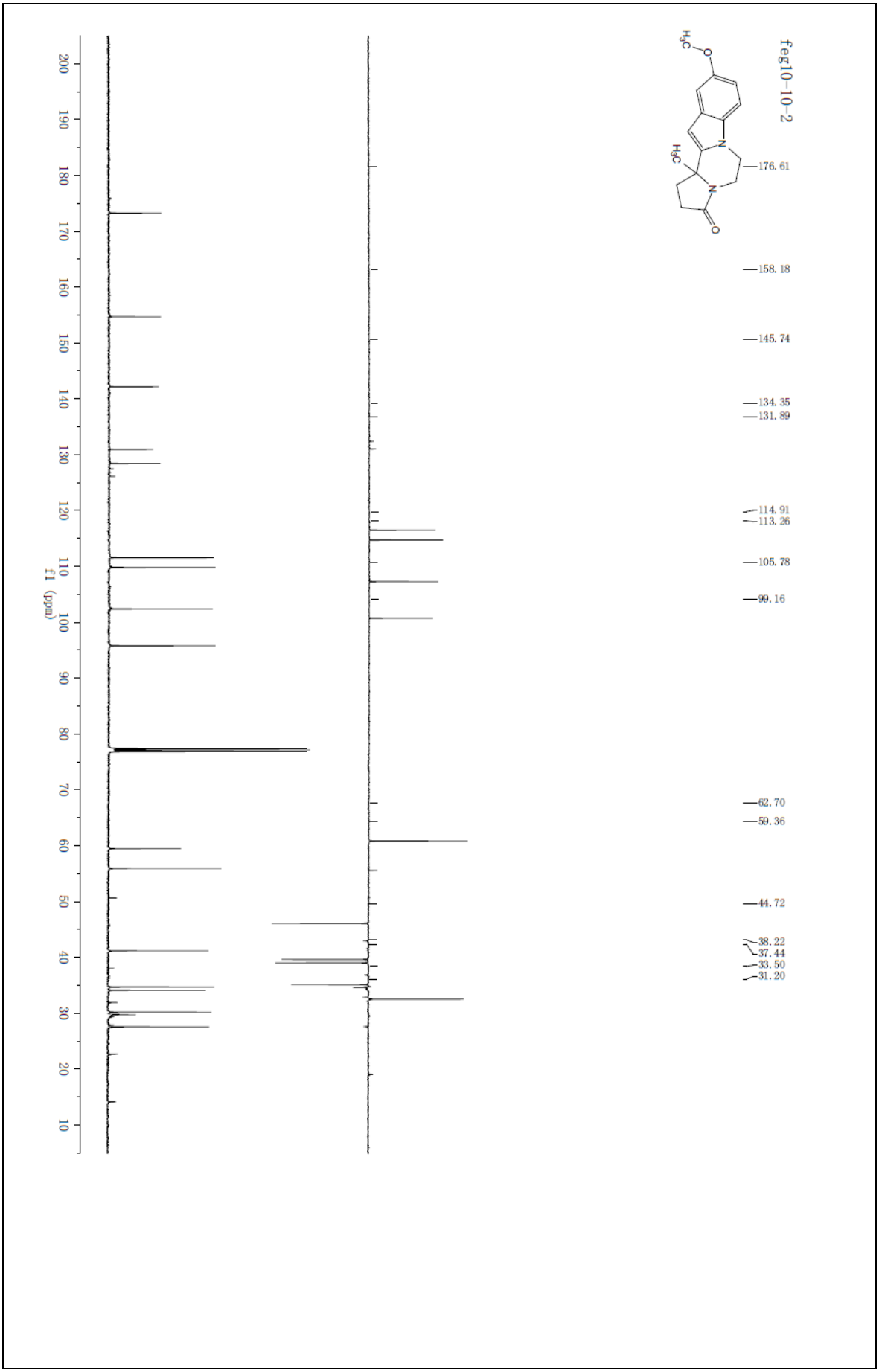
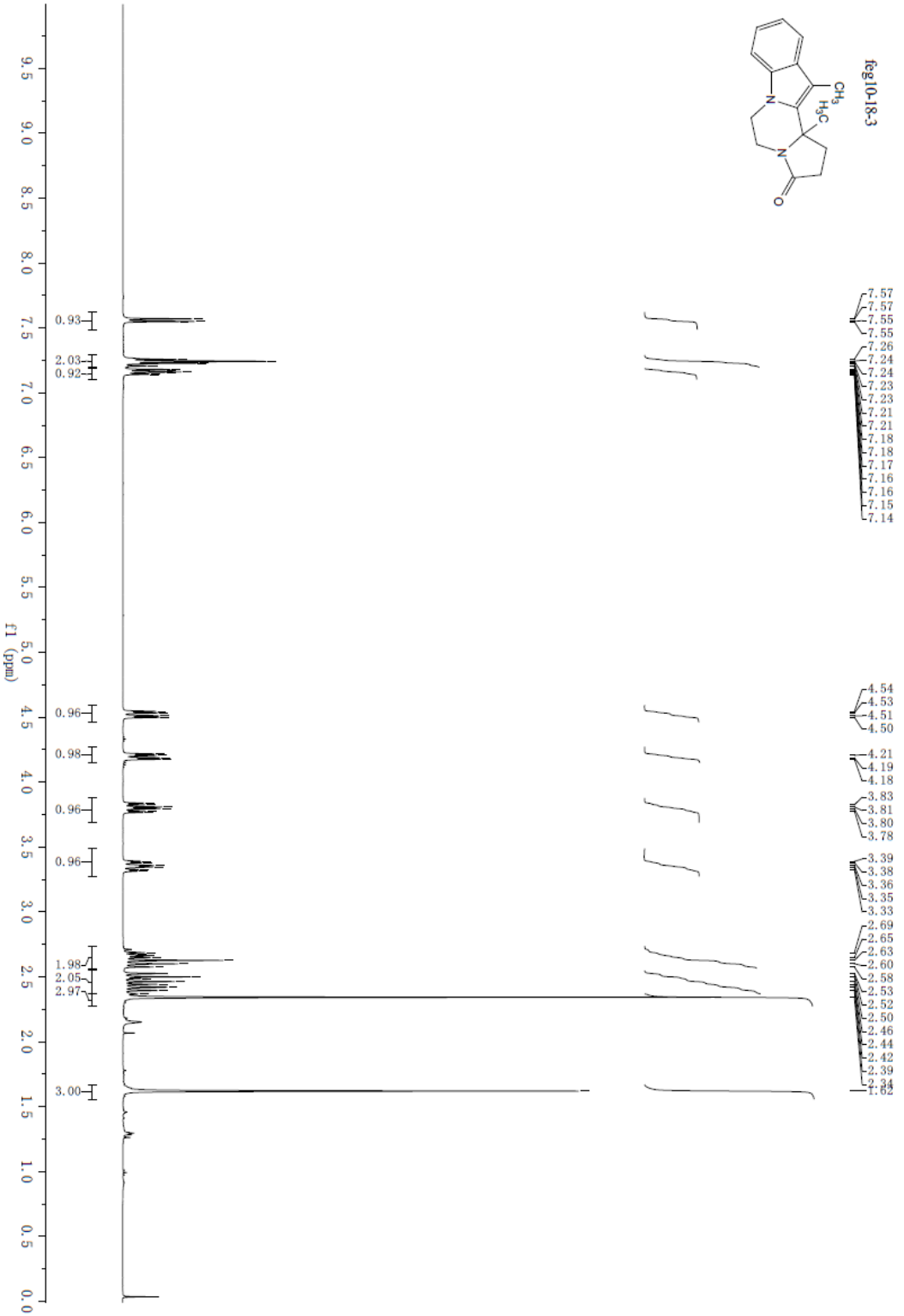
34.60
30.08
27.61
25.89
16.34

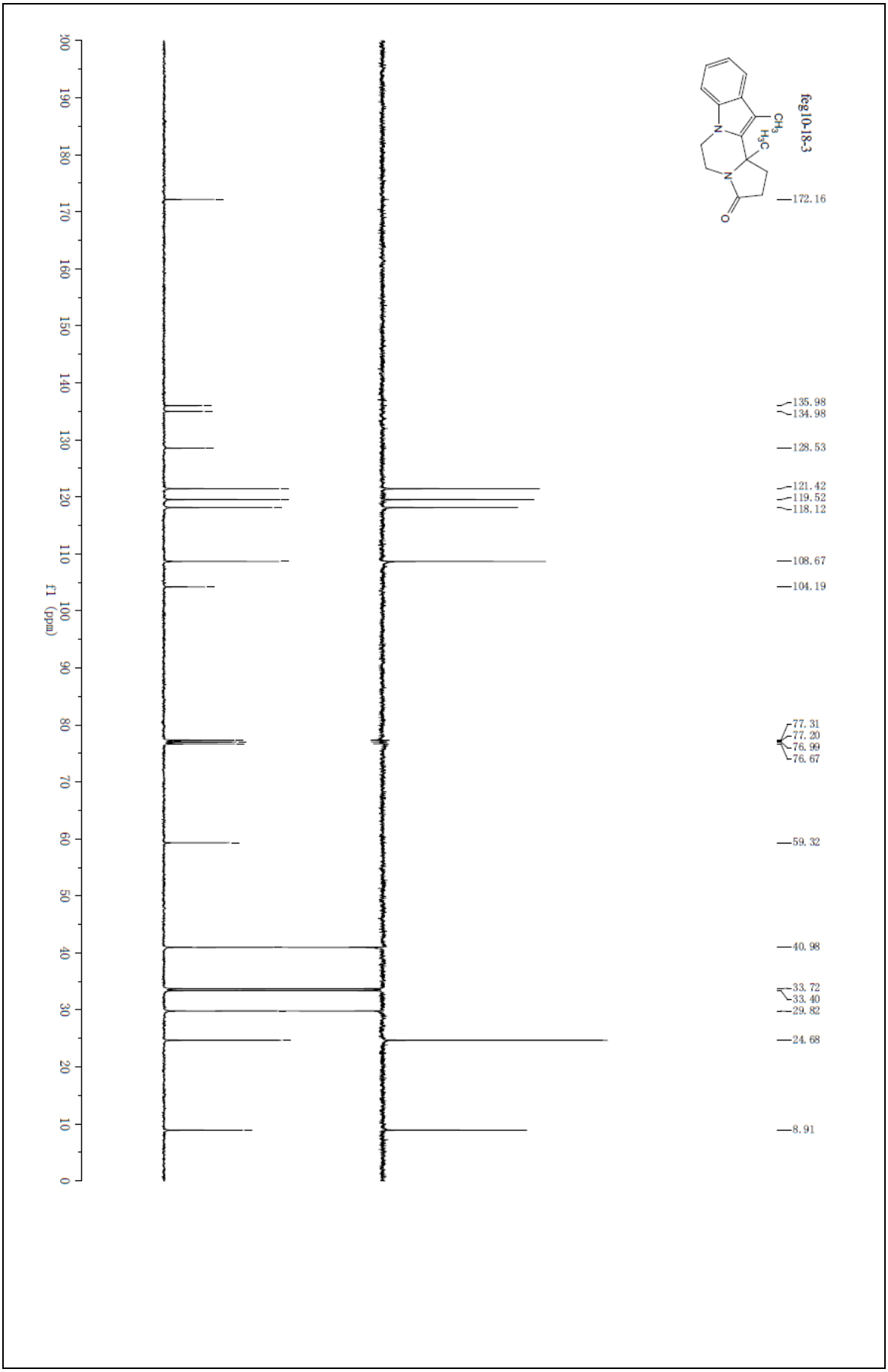
Compound 3f



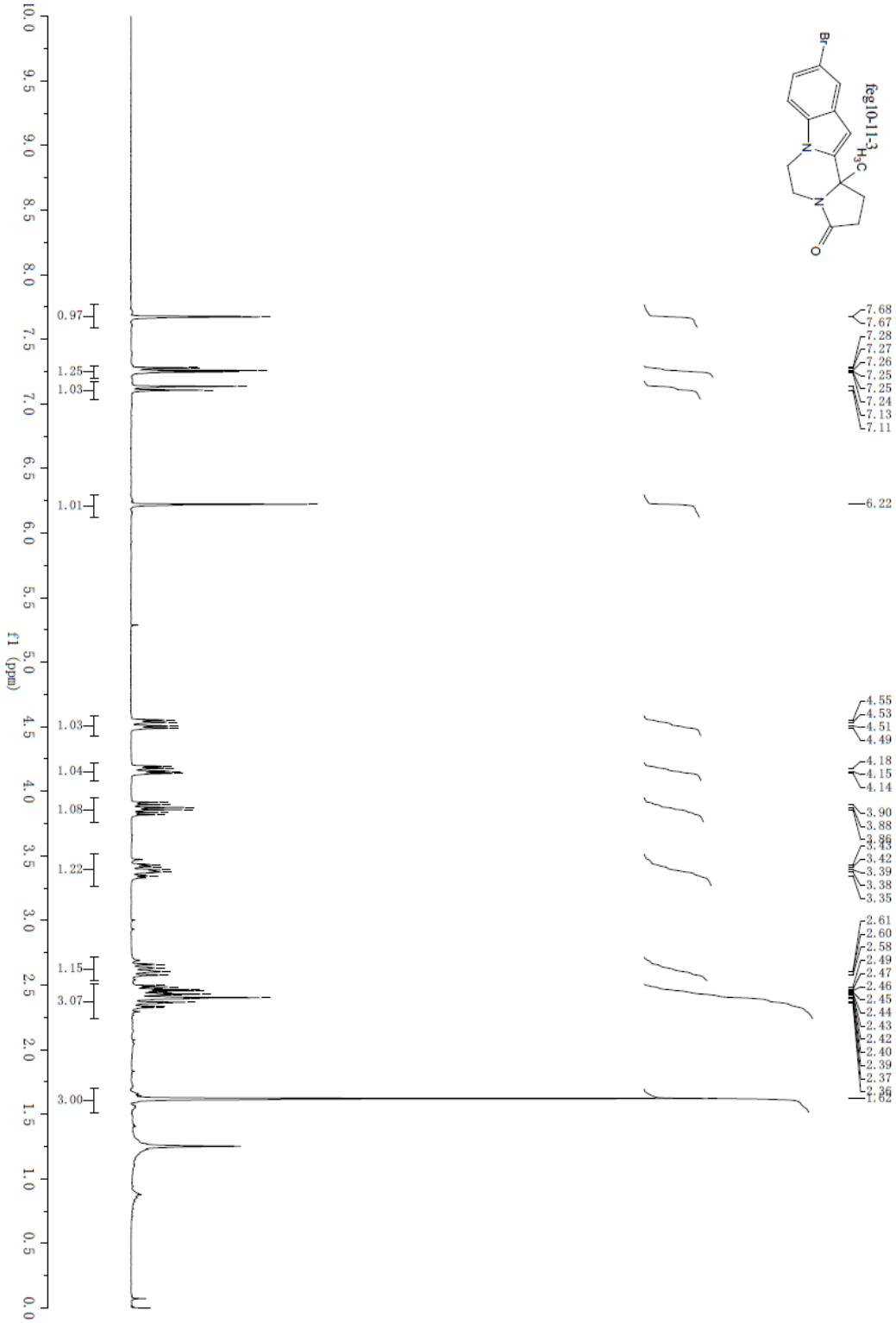


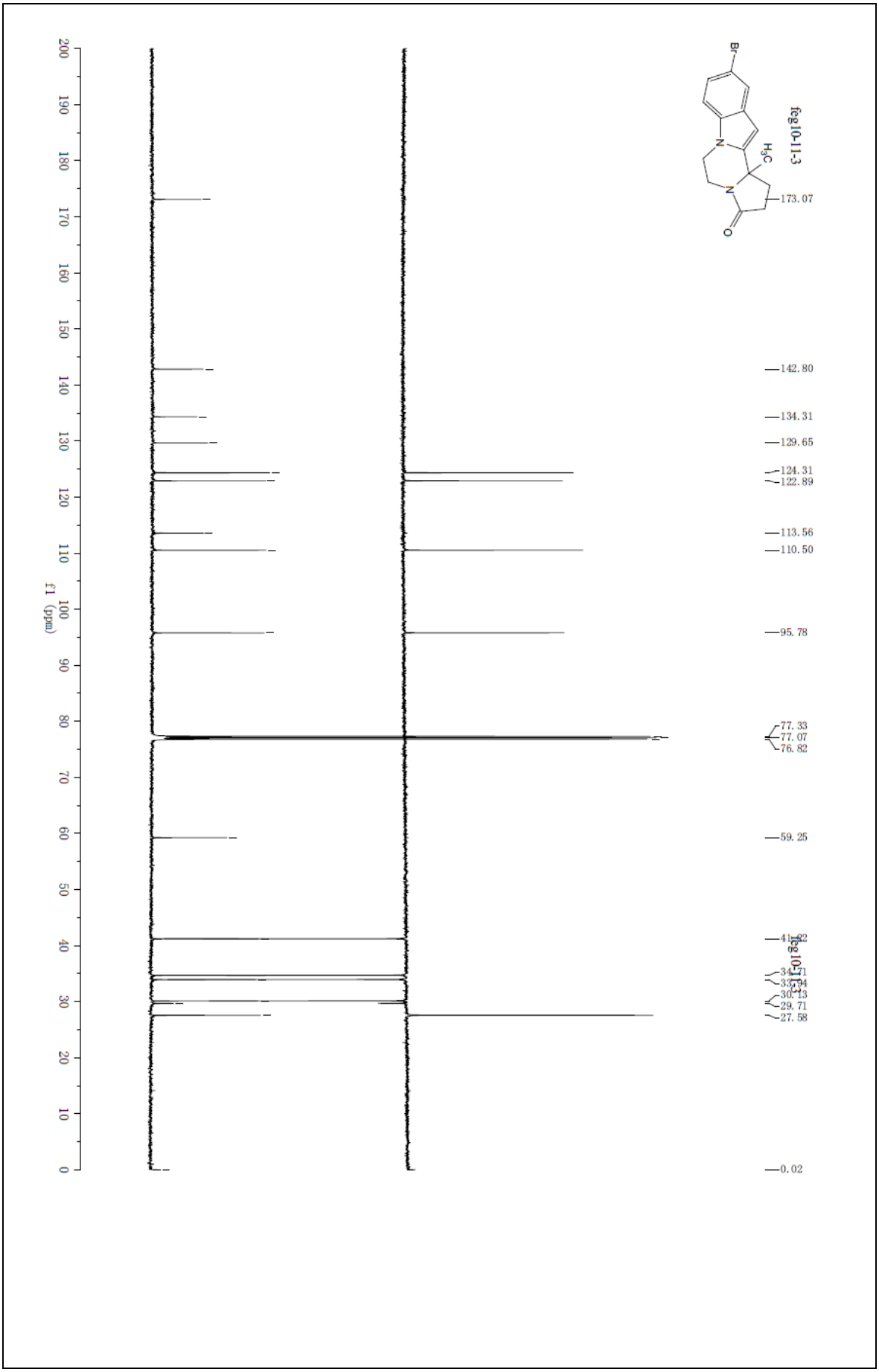
Compound 3g



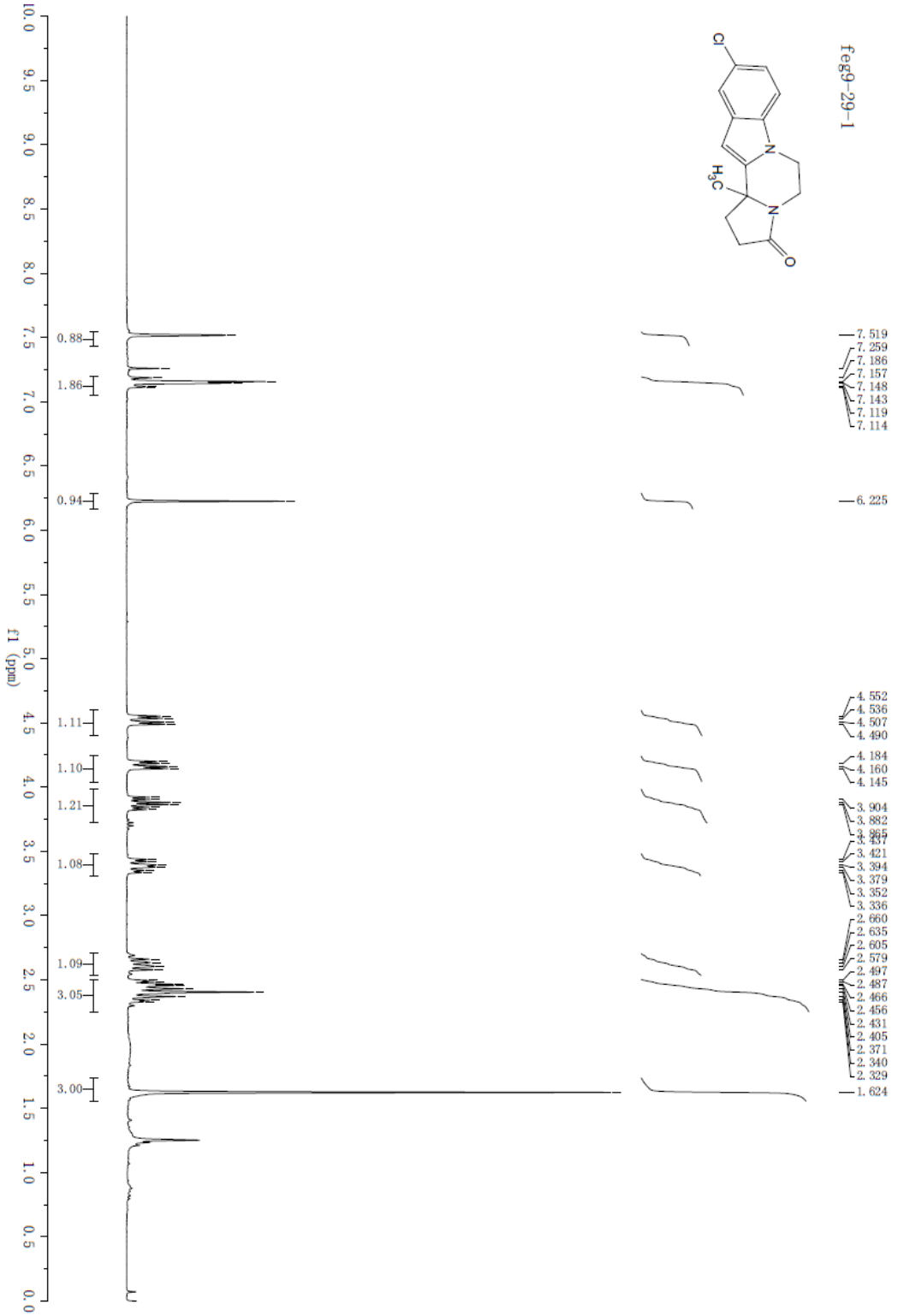

feg10-18-3
172.16
135.98
134.98
128.53
121.42
119.52
118.12
108.67
104.19
77.31
77.20
76.99
76.67
59.32
40.98
33.72
33.40
29.82
24.68
8.91
f1 (ppm)

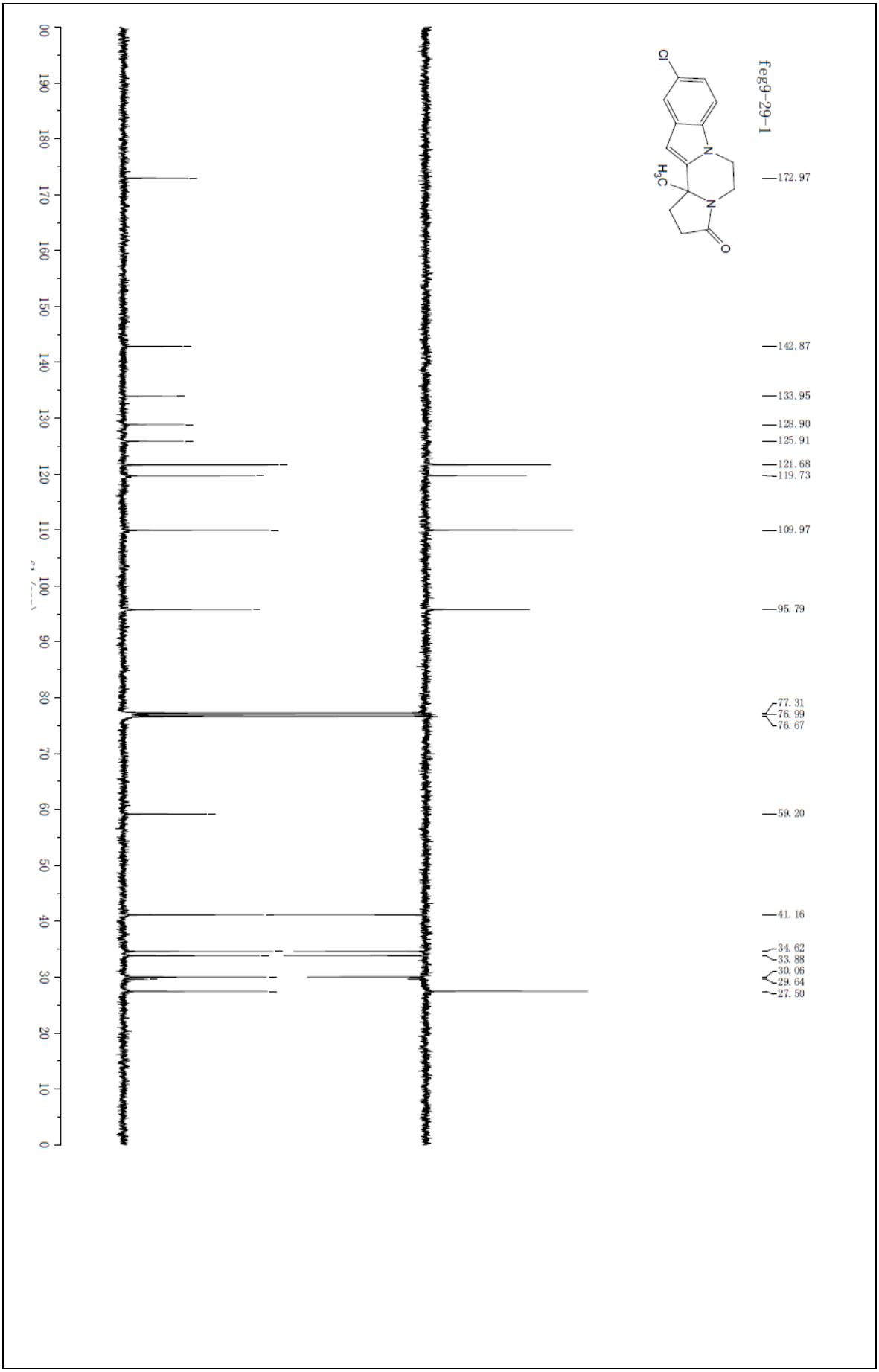
Compound 3h




feg10-11-3
Br
N
N
O
H3C
173.07
142.80
134.31
129.65
124.31
122.89
113.56
110.50
95.78
77.33
77.07
76.82
59.25
41.82
34.71
33.44
30.13
29.71
27.58
0.02
f1 (ppm)

Compound 3i





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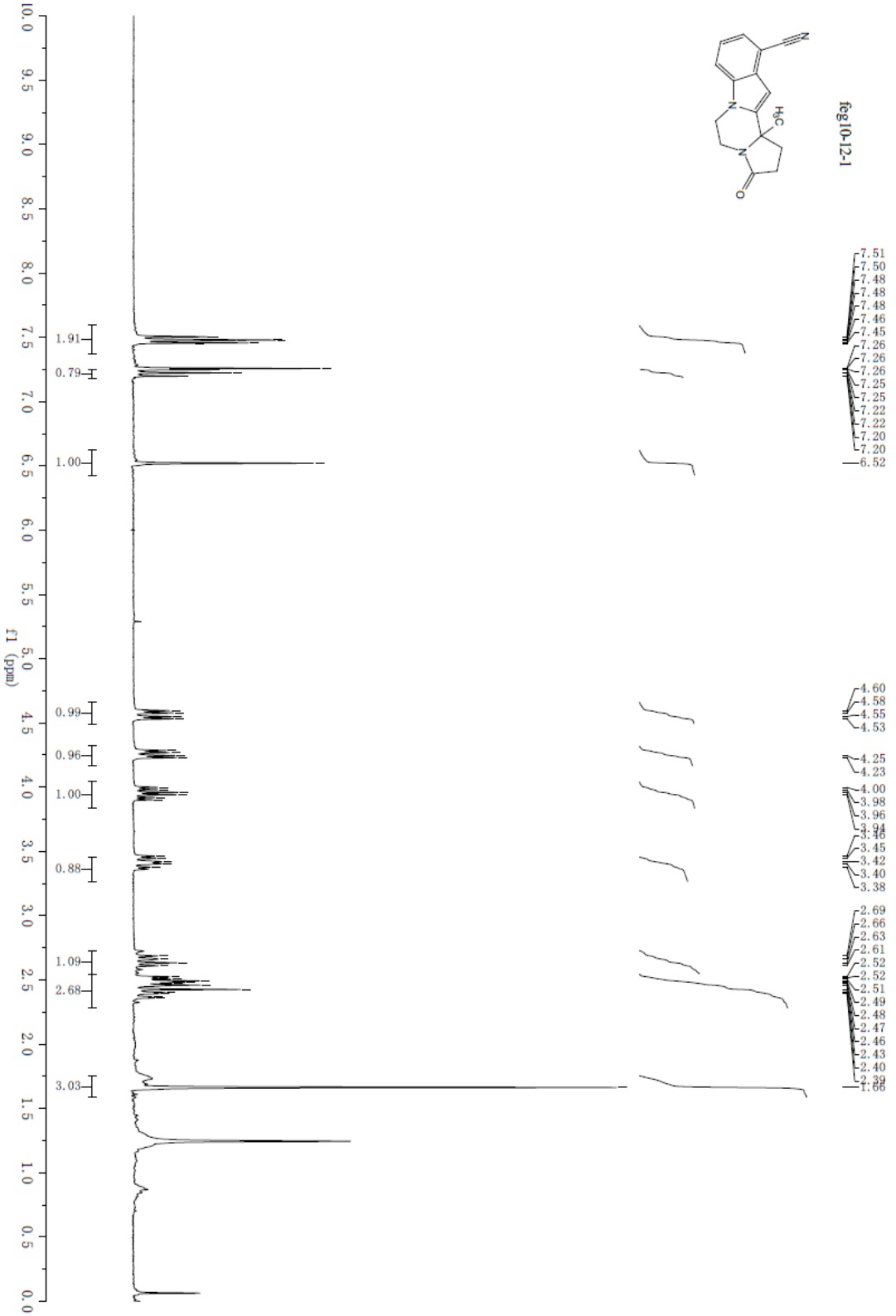

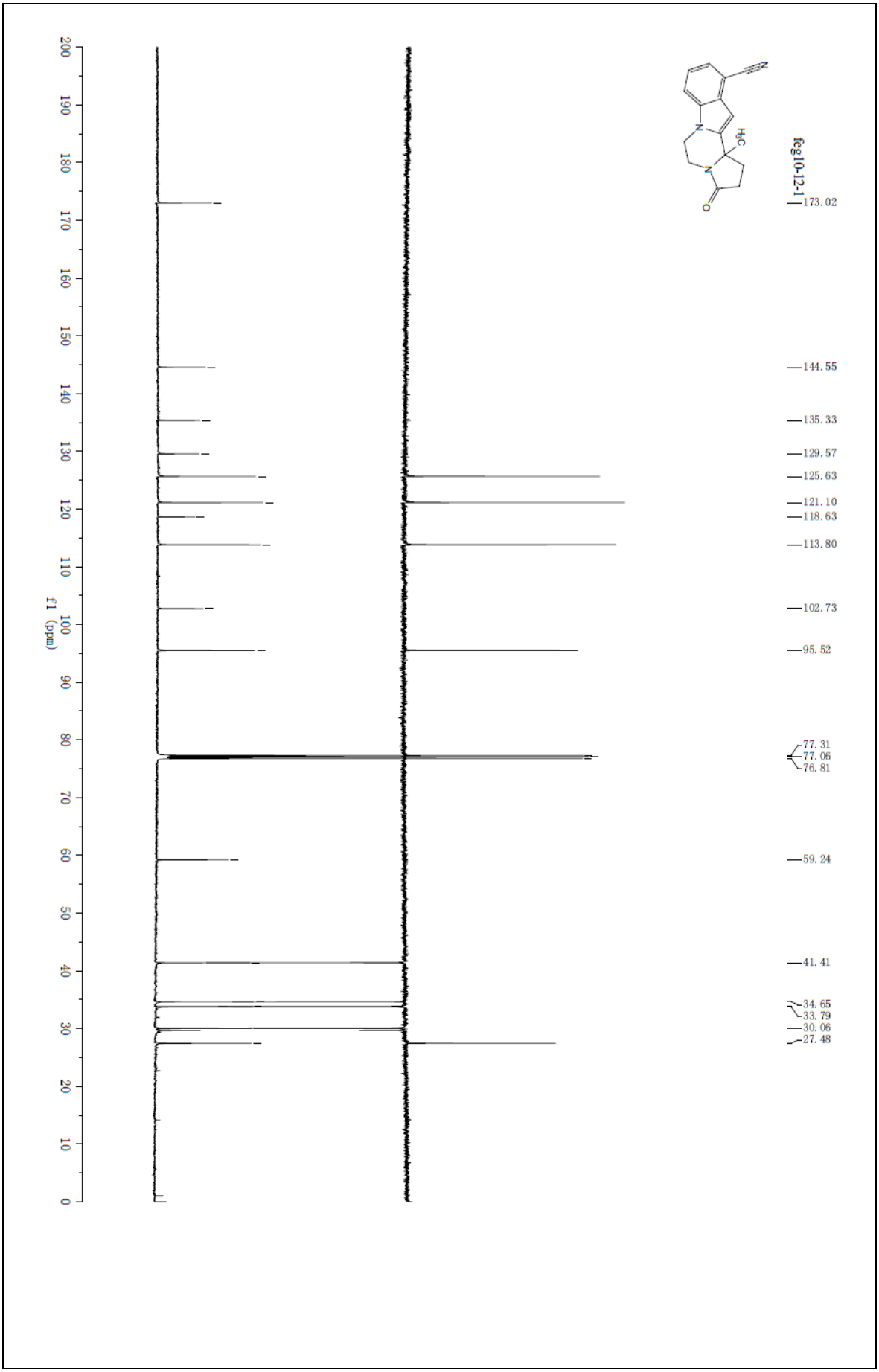

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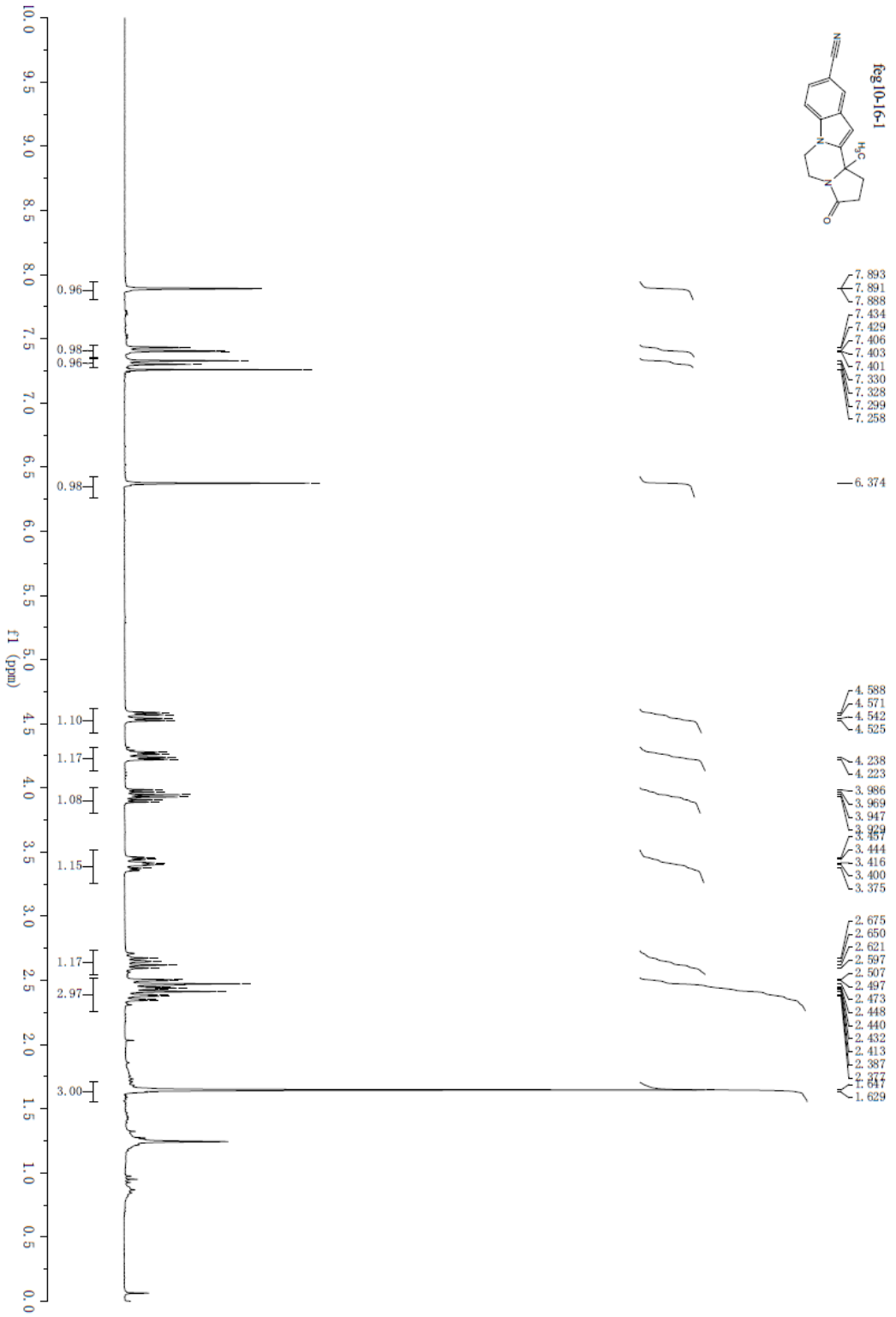
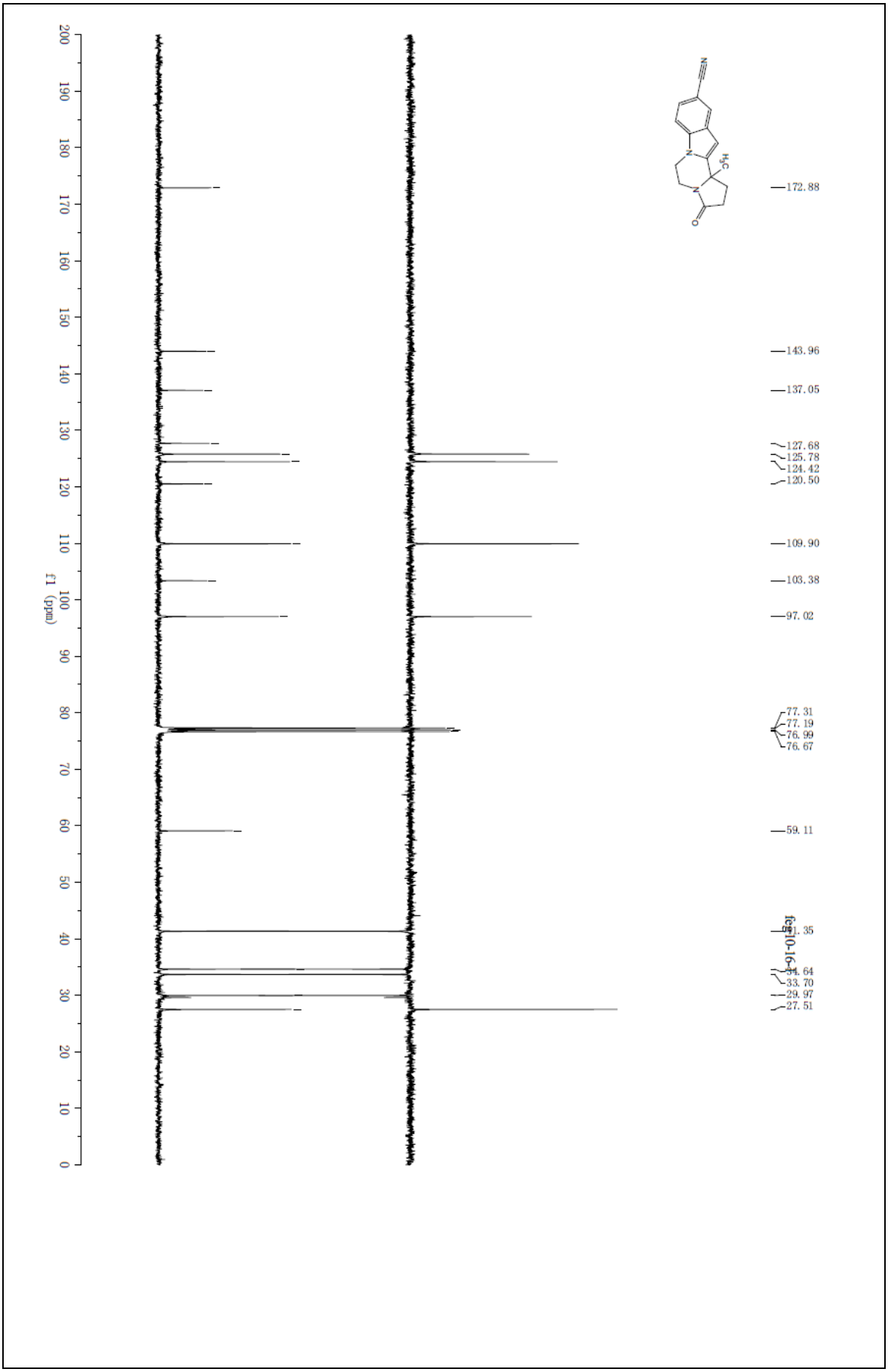
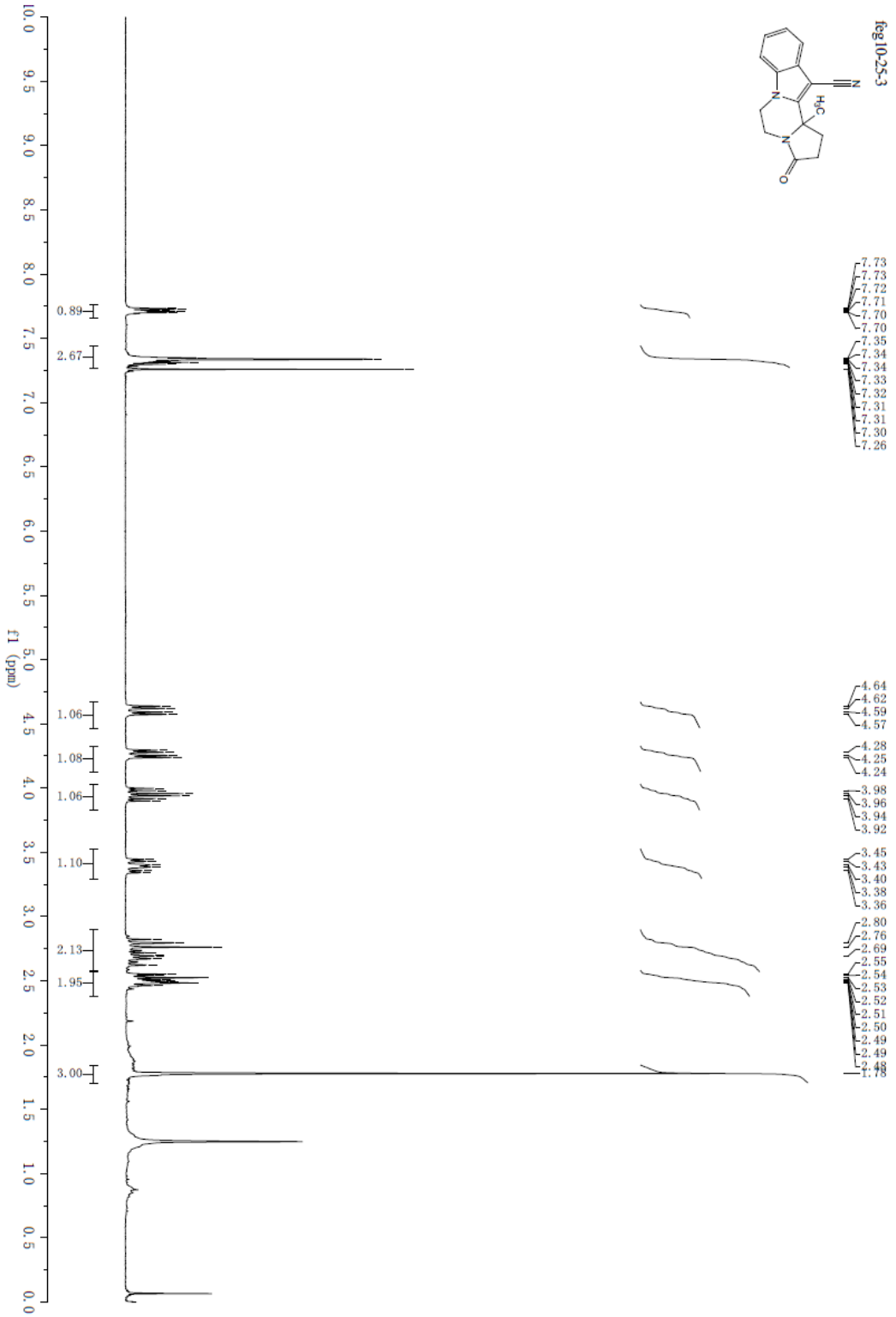
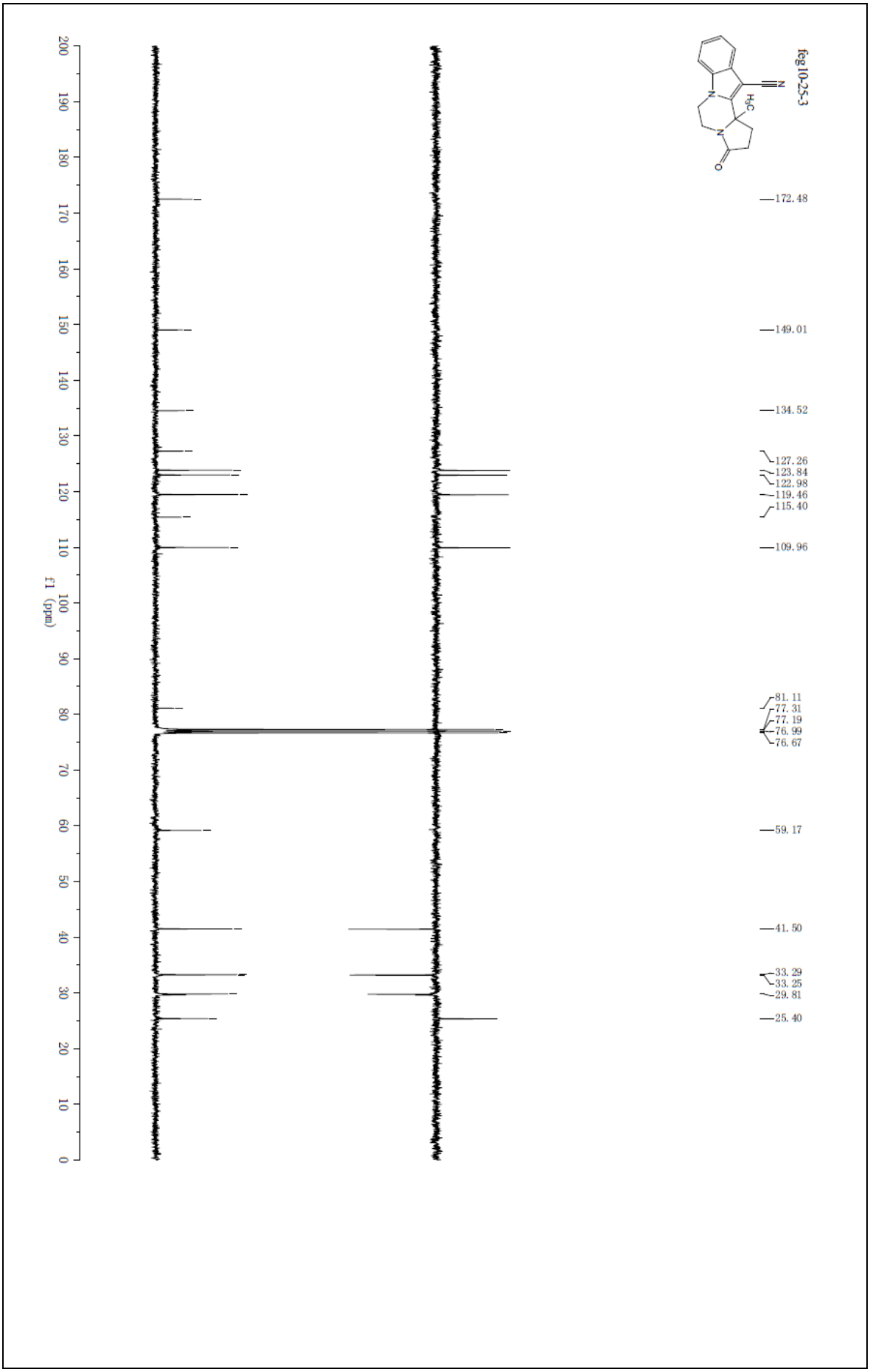




Compound 3l

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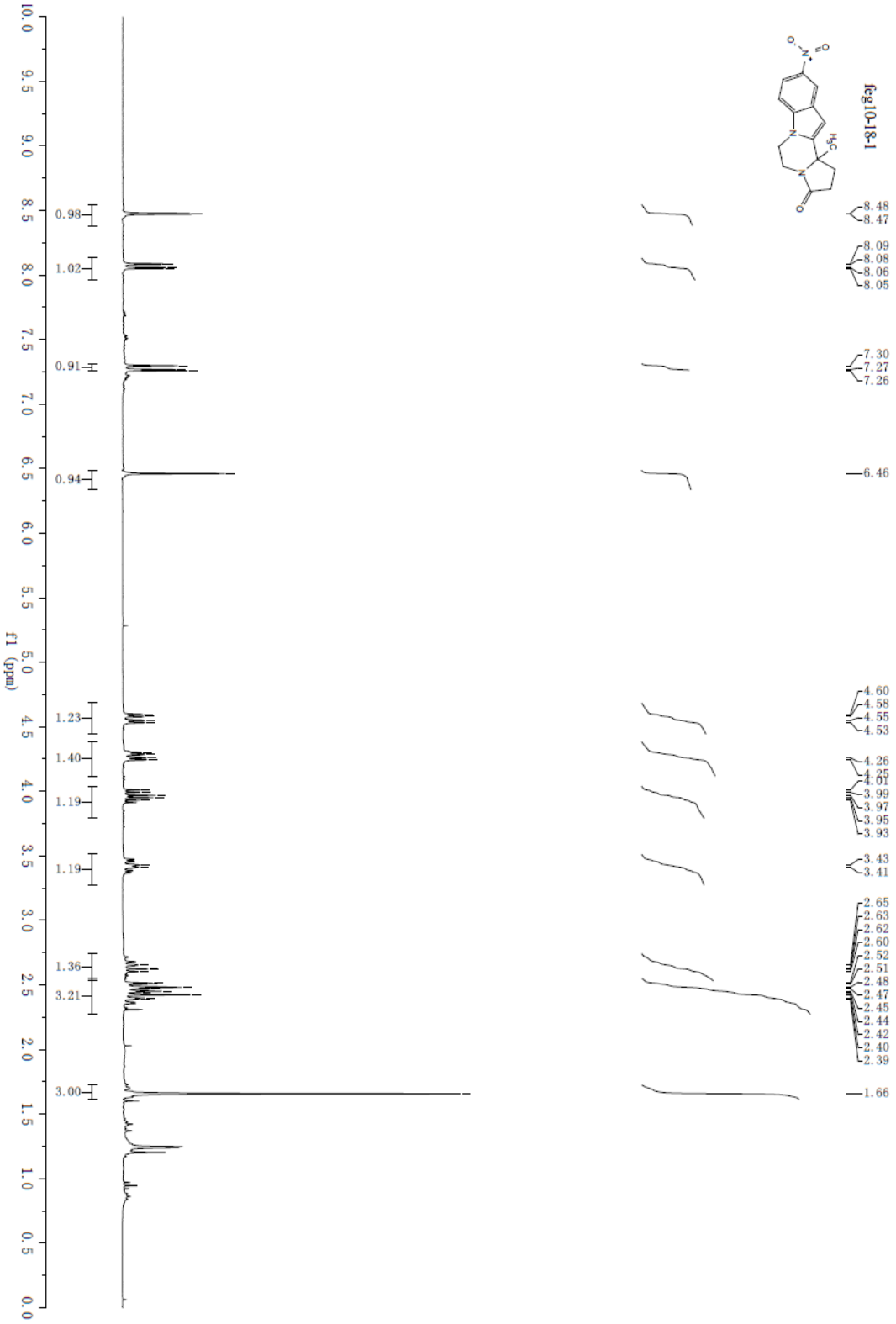
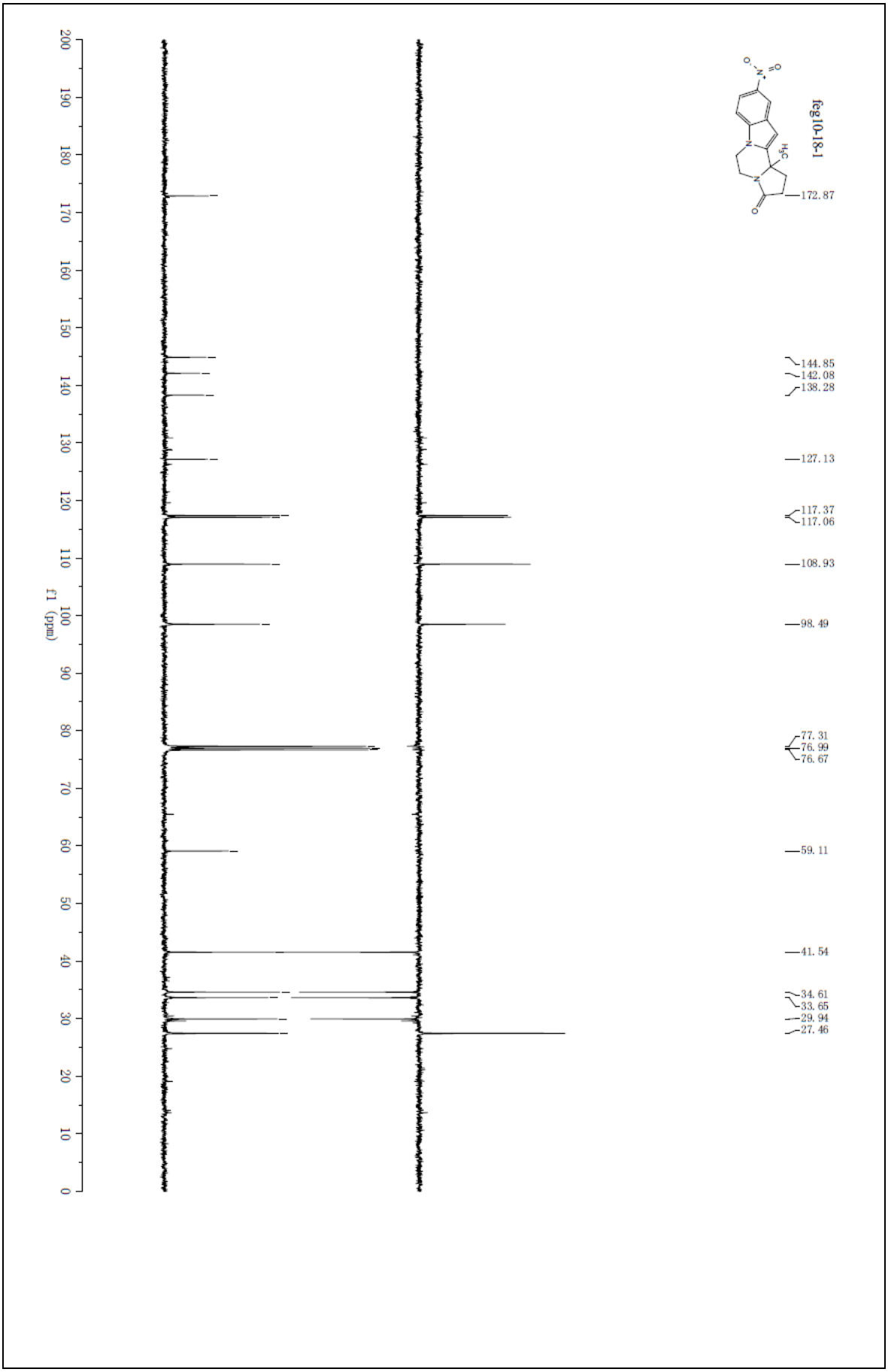
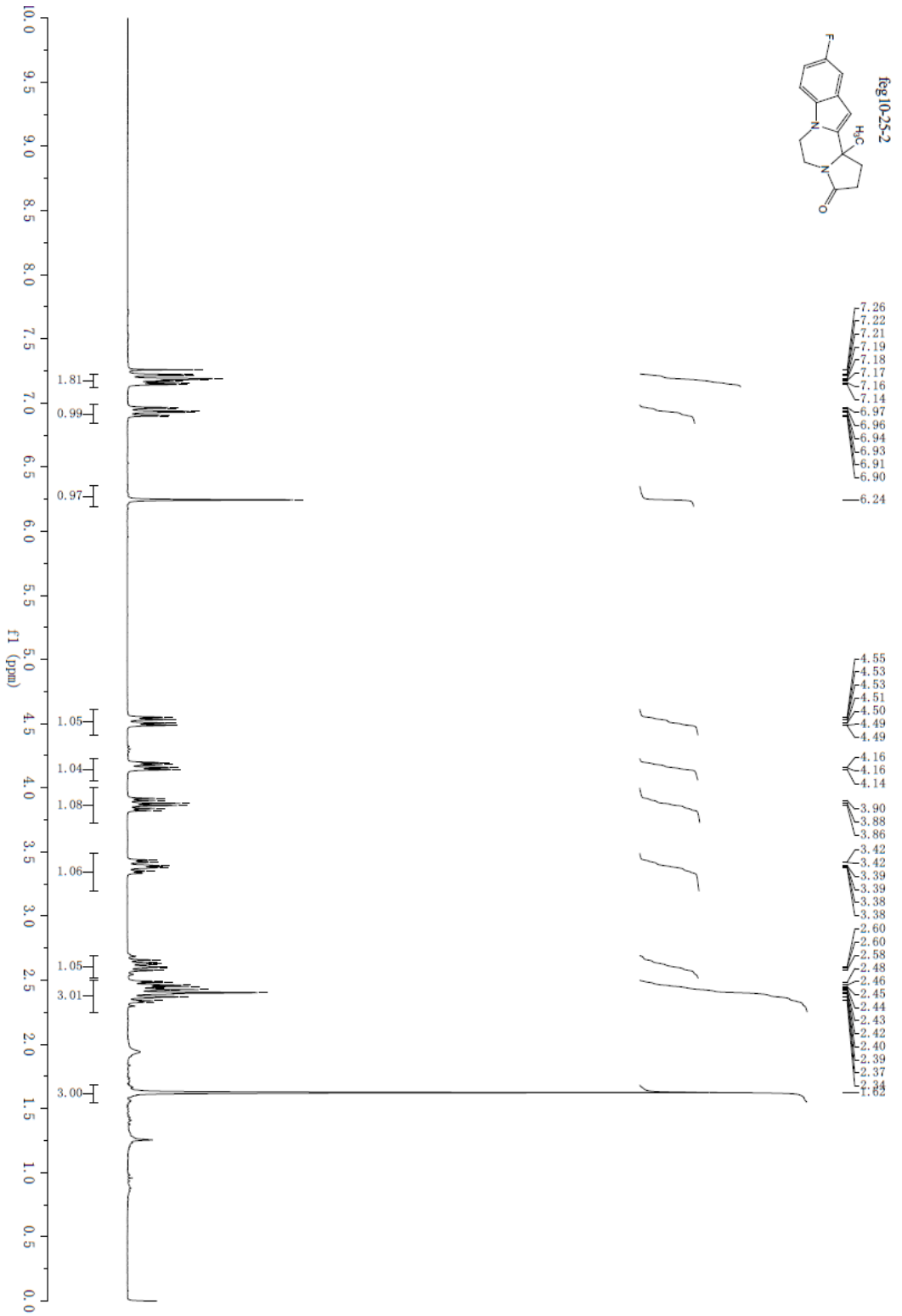
172.48
149.01
134.52
127.26
123.84
122.98
119.46
115.40
109.96
81.11
77.31
77.19
76.99
76.67
59.17
41.50
33.29
33.25
29.81
25.40
f1 (ppm)

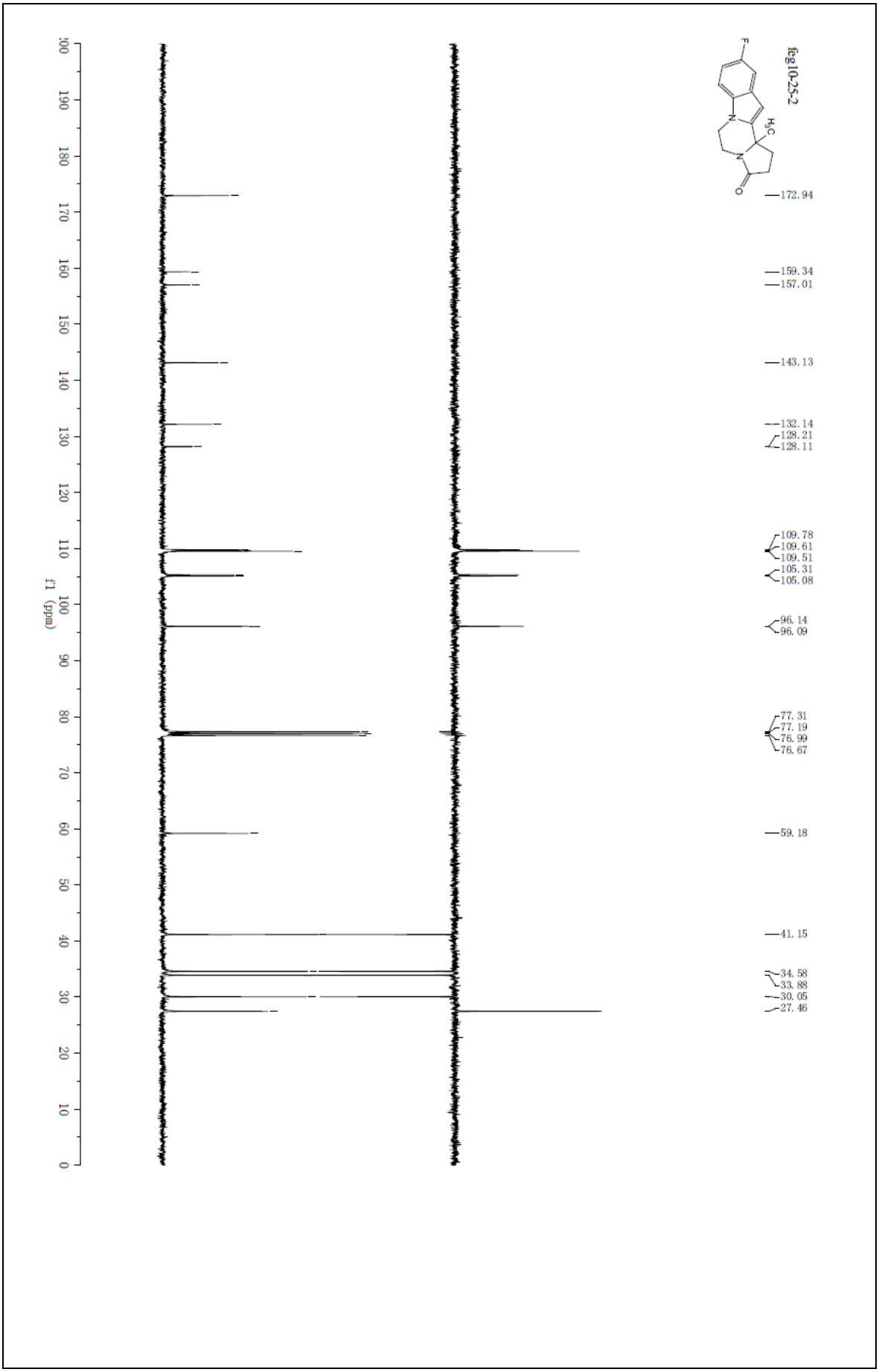
Compound 3m



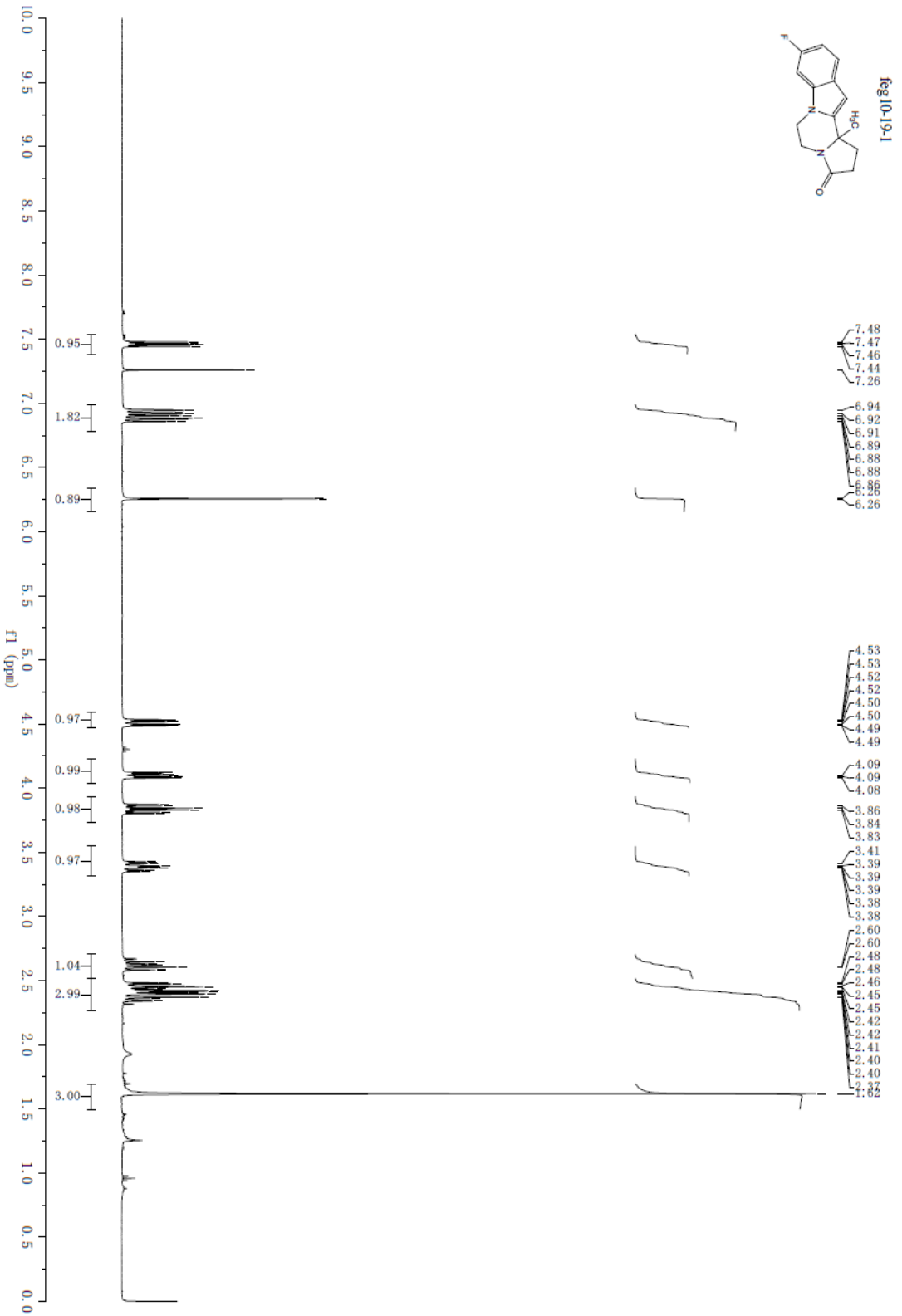


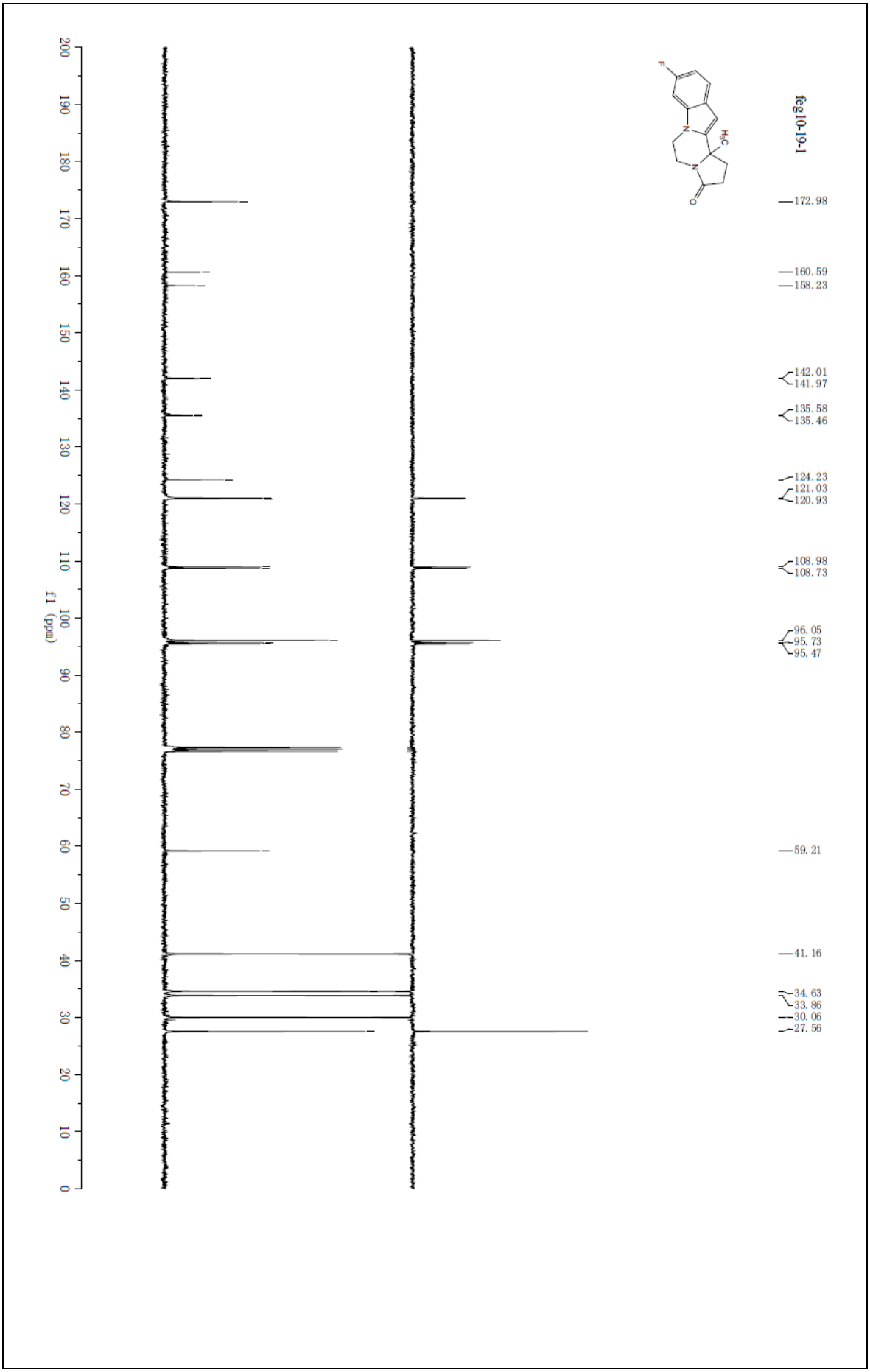
Compound 3n




fcg10-25-2
F
H3C
N
N
O
172.94
159.34
157.01
143.13
132.14
128.21
128.11
109.78
109.61
109.51
105.31
105.08
96.14
96.09
77.31
77.19
76.99
76.67
59.18
41.15
34.58
33.88
30.05
27.46
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Compound 3o

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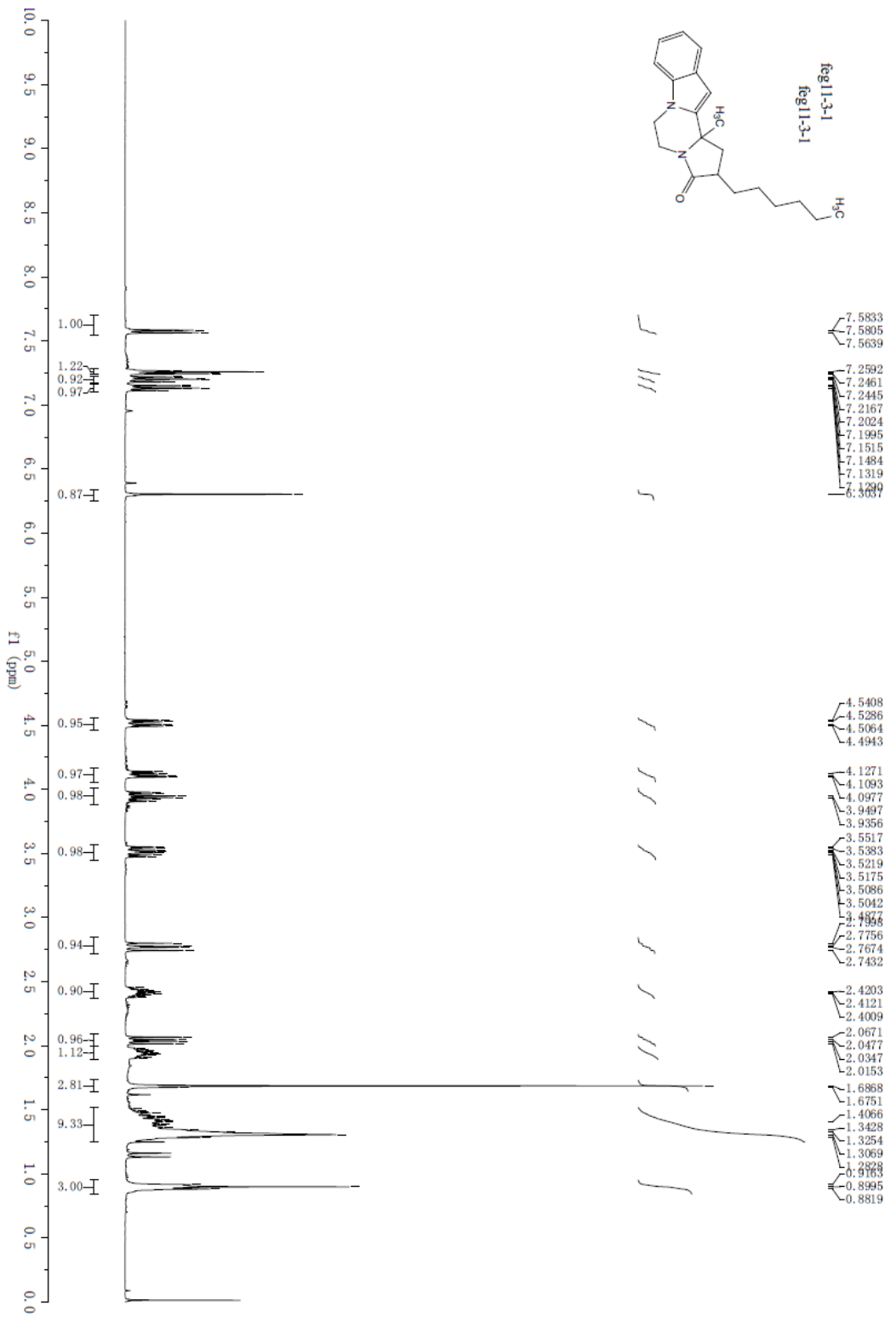
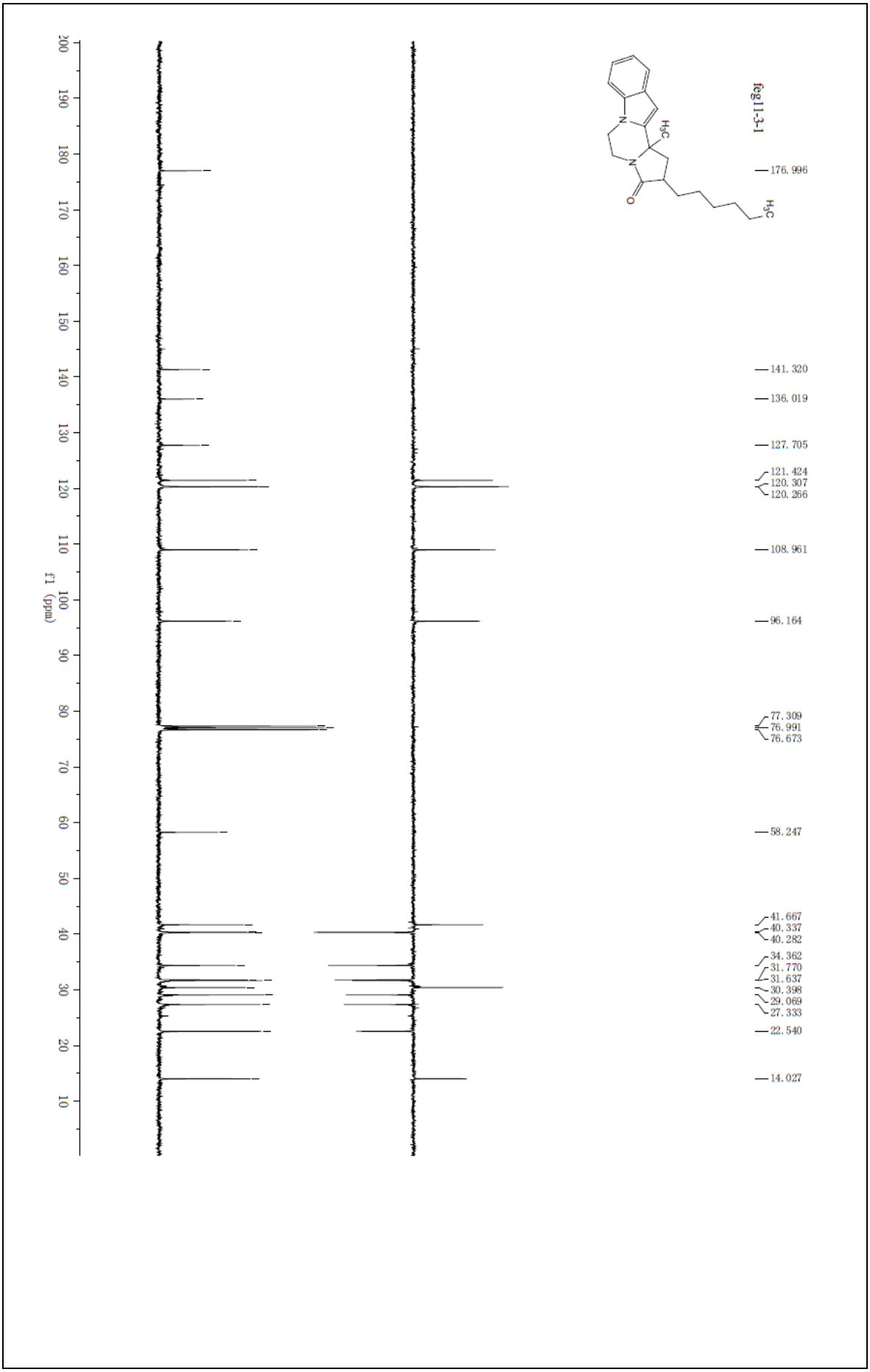
160.59
158.23
142.01
141.97
135.58
135.46
124.23
121.03
120.93
108.98
108.73
96.05
95.73
95.47
59.21
41.16
34.63
33.86
30.06
27.56
f1 (ppm)

Compound 3p




jea11-3-1
176.996
141.320
136.019
127.705
121.424
120.307
120.266
108.961
96.164
77.309
76.991
76.673
58.247
41.667
40.337
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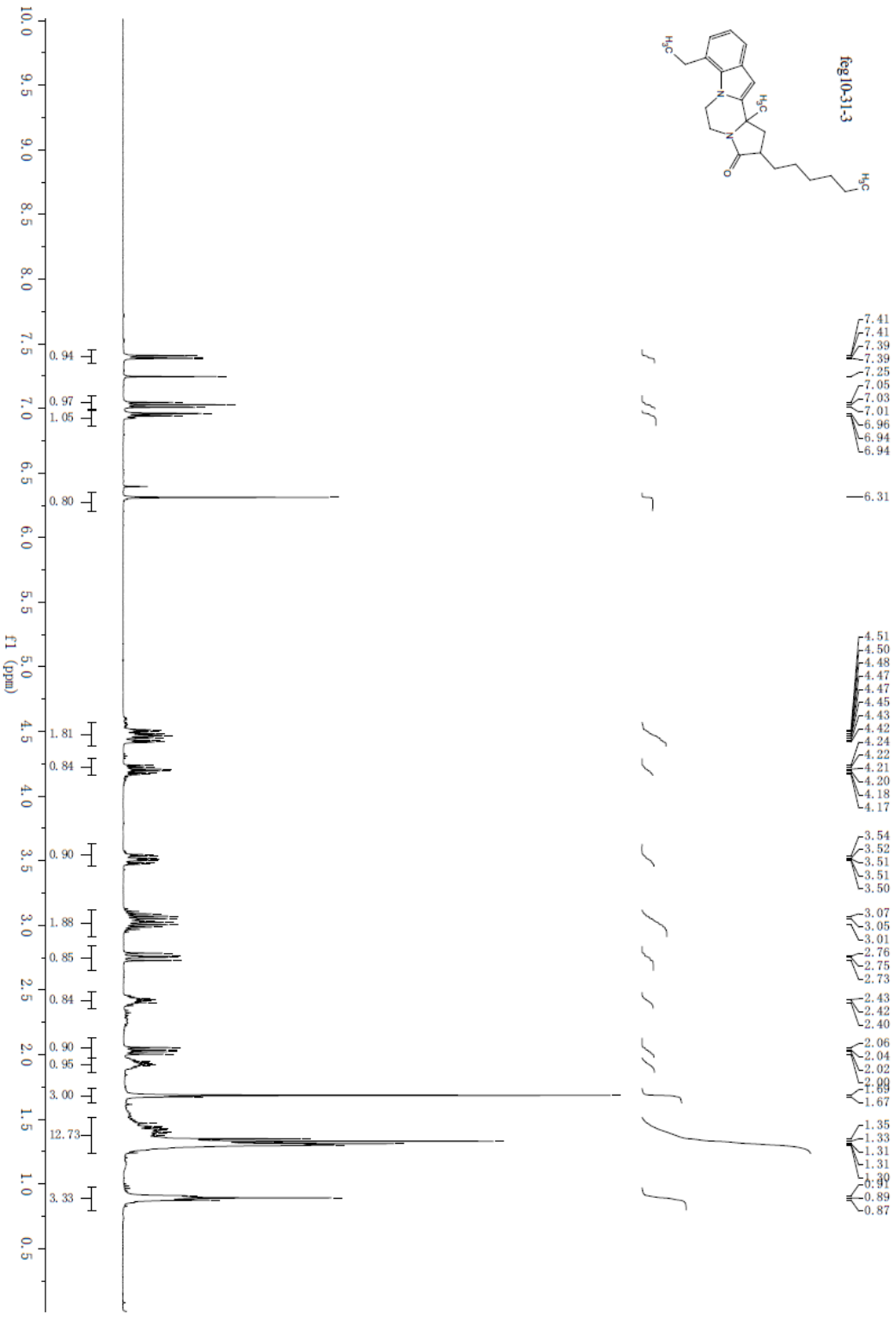
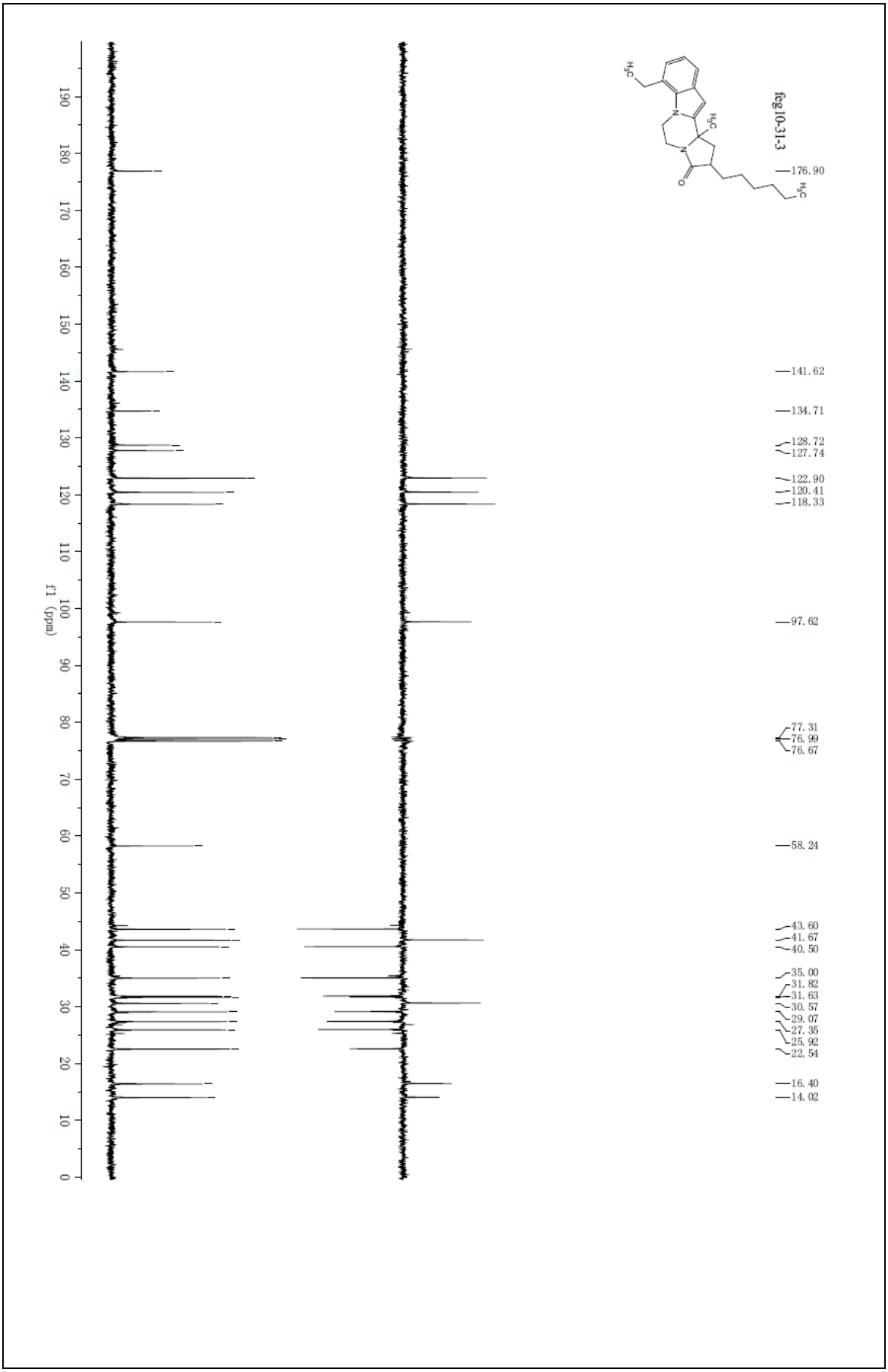
31.770
31.637
30.398
29.069
27.333
22.540
14.027
f1 (ppm)

Compound 3q

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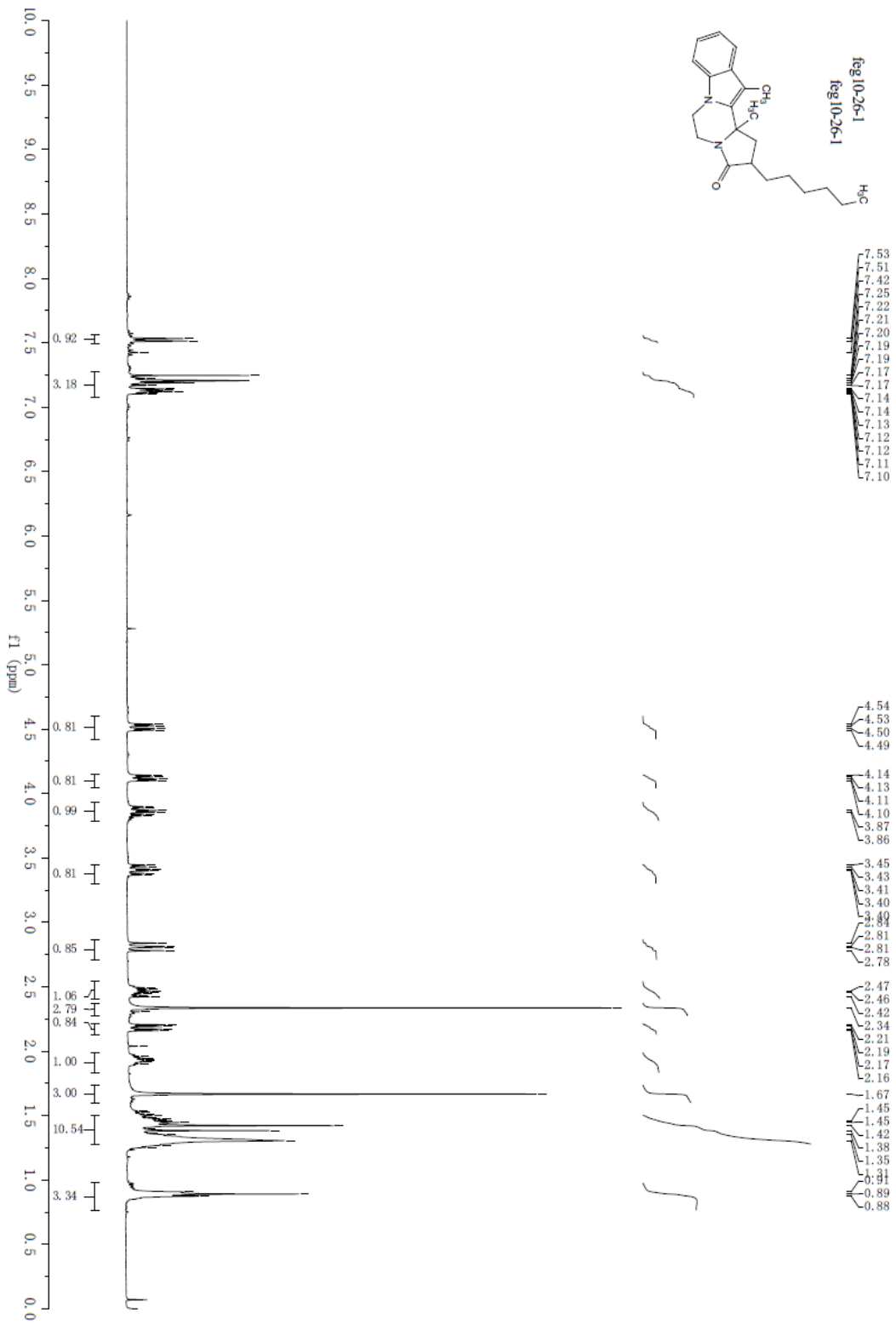
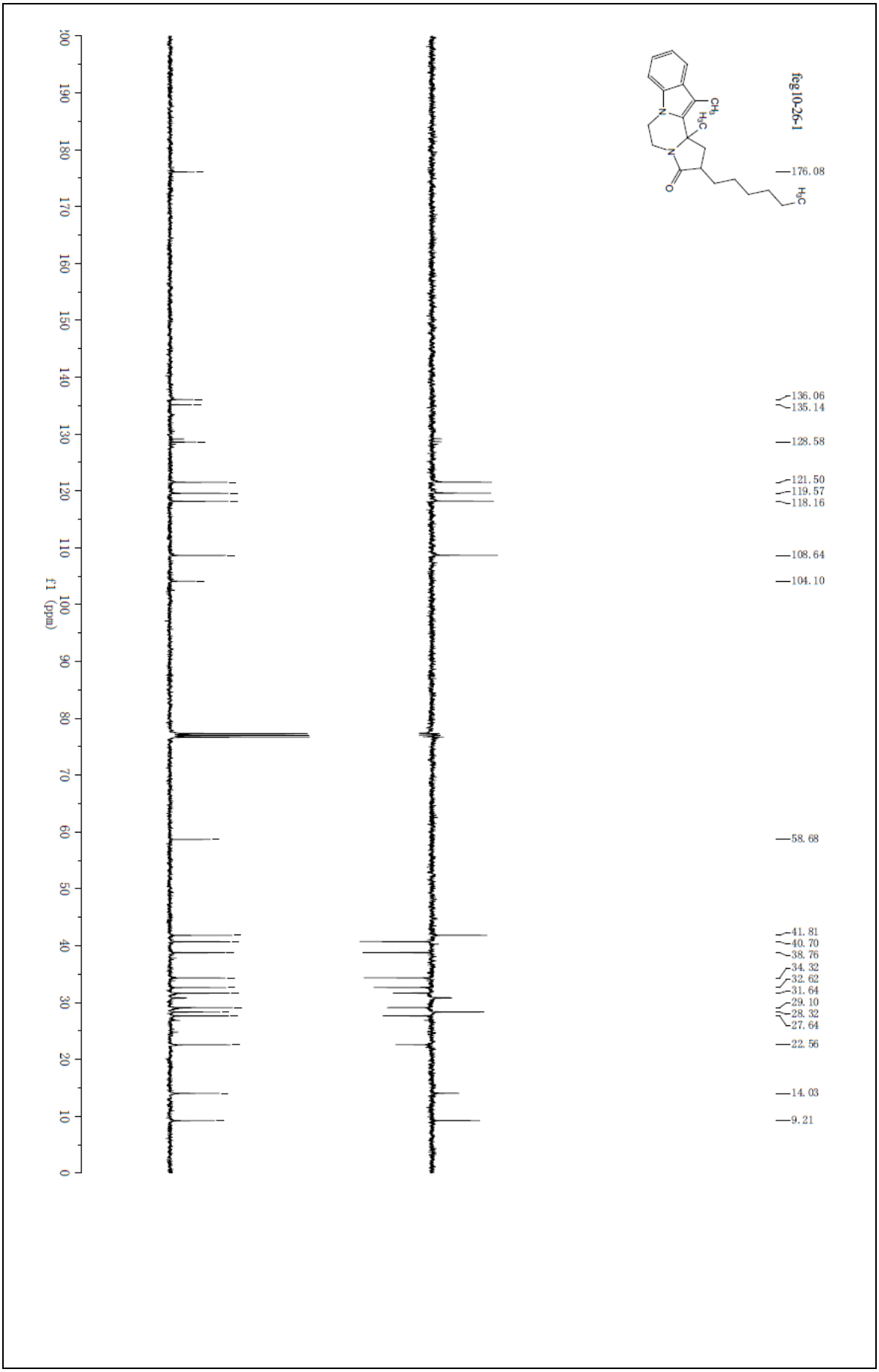
141.62
134.71
128.72
127.74
122.90
120.41
118.33
97.62
77.31
76.99
76.67
58.24
43.60
41.67
40.50
35.00
31.82
31.63
30.57
29.07
27.35
25.92
22.54
16.40
14.02
f1 (ppm)

Compound 3r




fea-10-26-1
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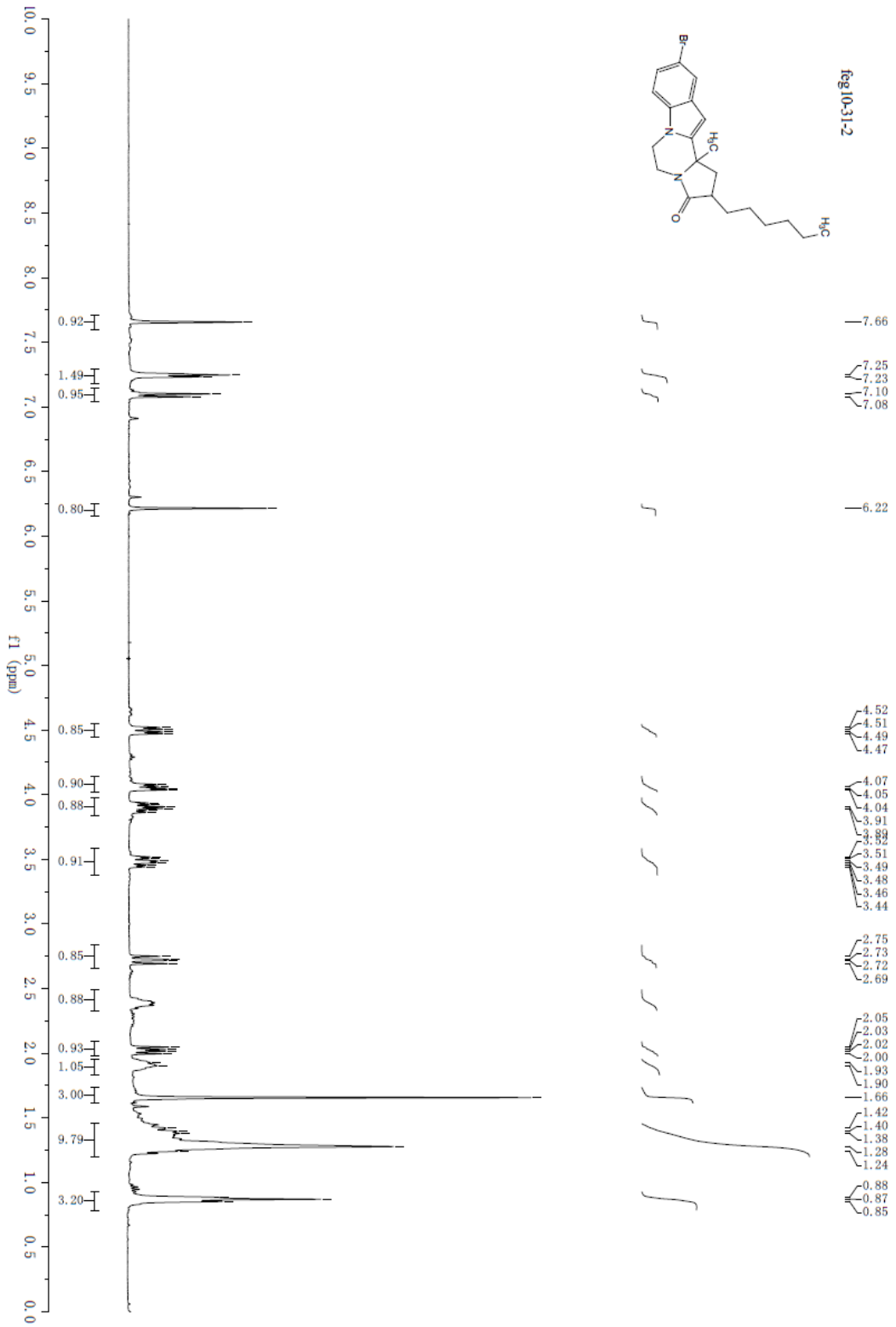
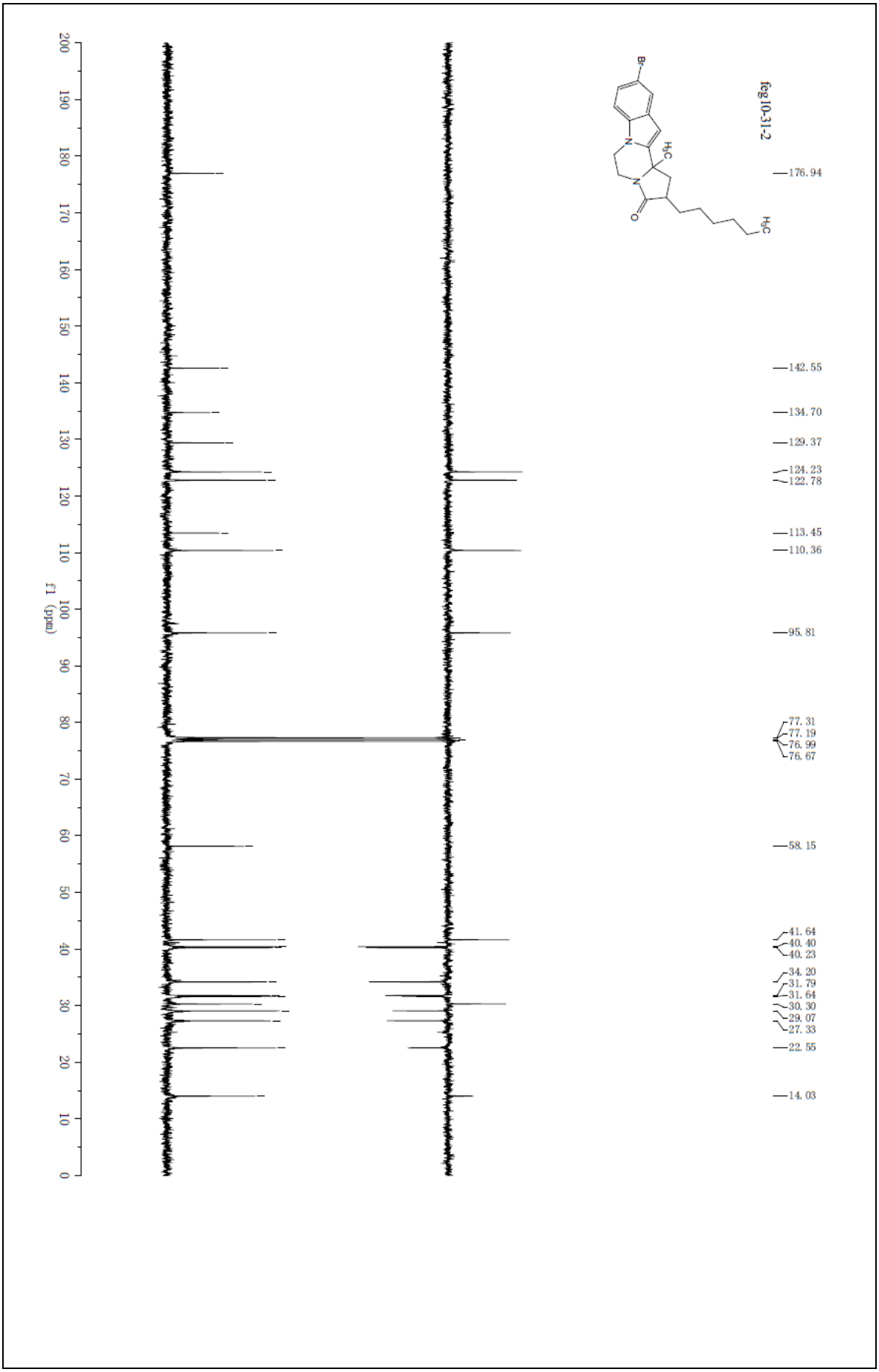
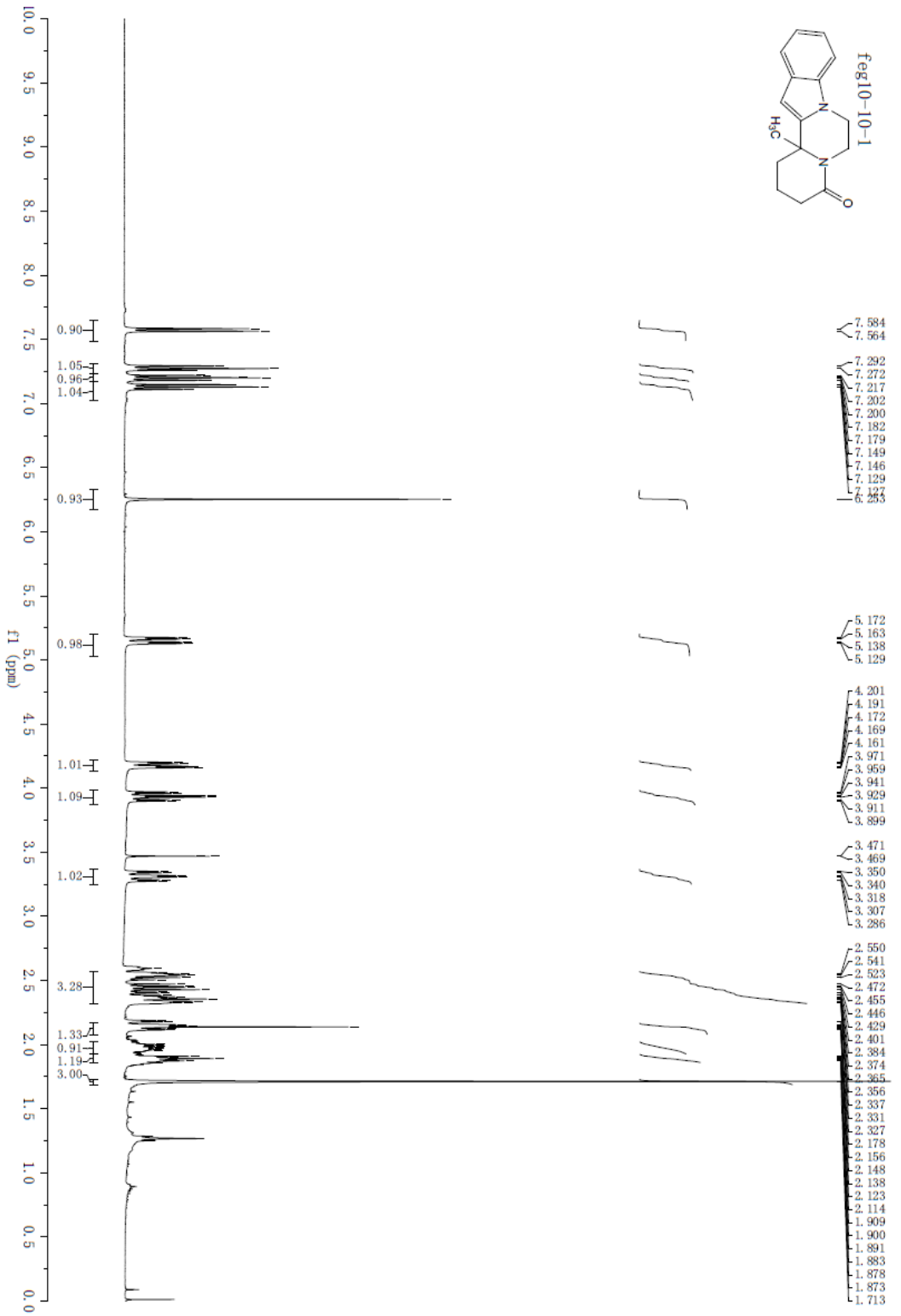
136.06
135.14
128.58
121.50
119.57
118.16
108.64
104.10
58.68
41.81
40.70
38.76
34.32
32.62
31.64
29.10
28.32
27.64
22.56
14.03
9.21
f1 (ppm)

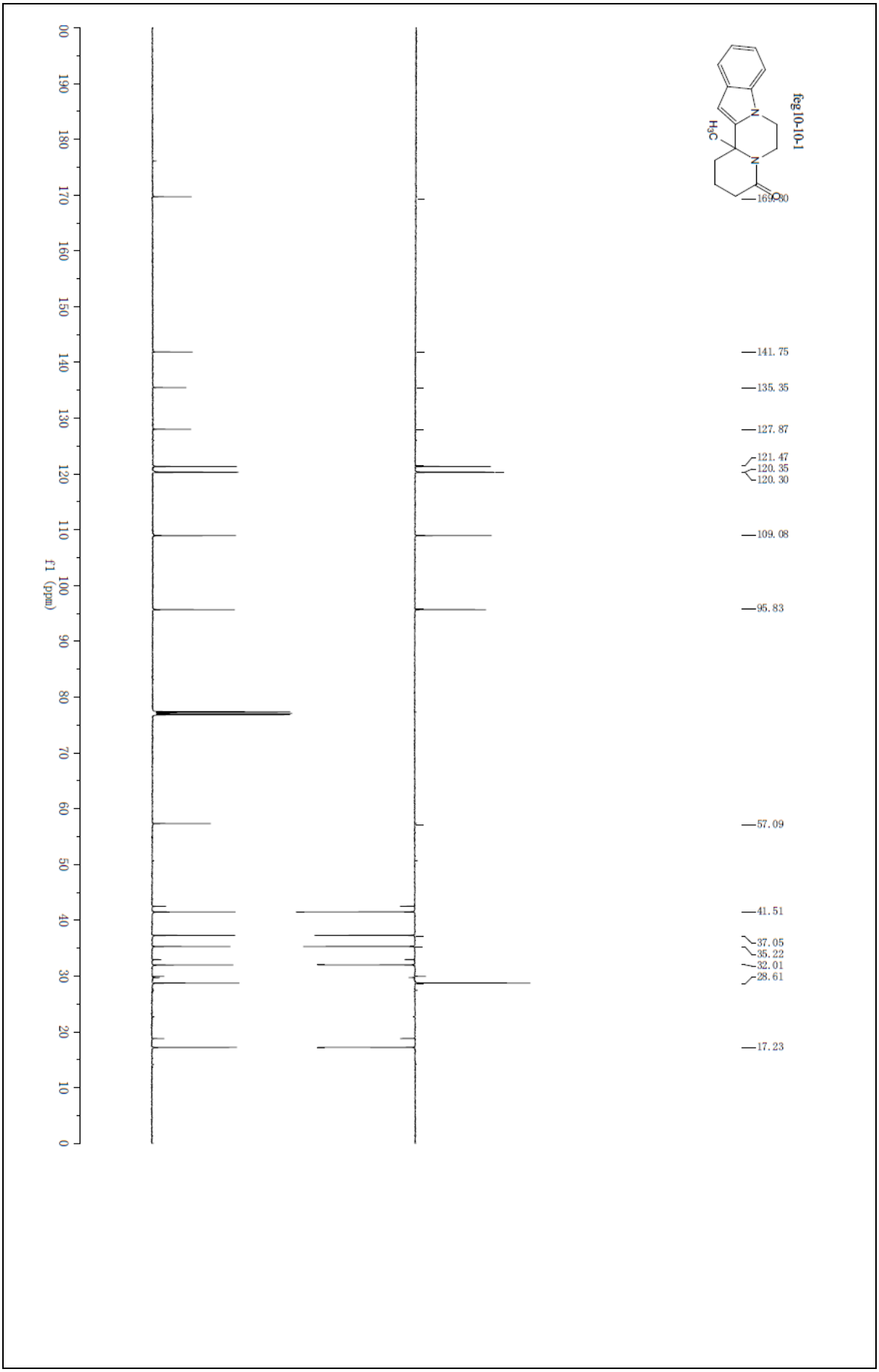
Compound 3s



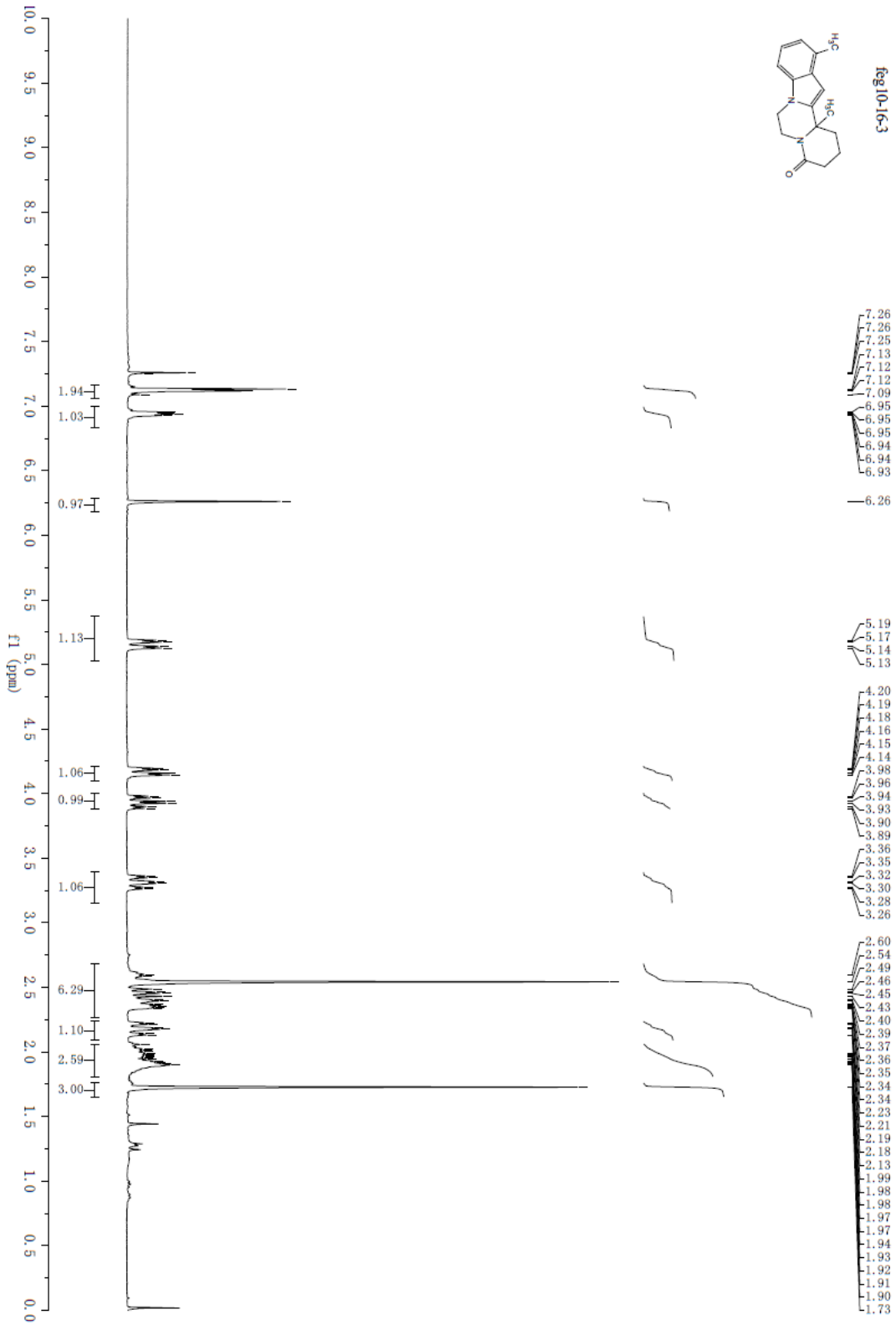

feg10-31-2
Br
N
N
O
H3C
H3C
f1 (ppm)
176.94
142.55
134.70
129.37
124.23
122.78
113.45
110.36
95.81
77.31
77.19
76.99
76.67
58.15
41.64
40.40
40.23
34.20
31.79
31.64
30.30
29.07
27.33
22.55
14.03

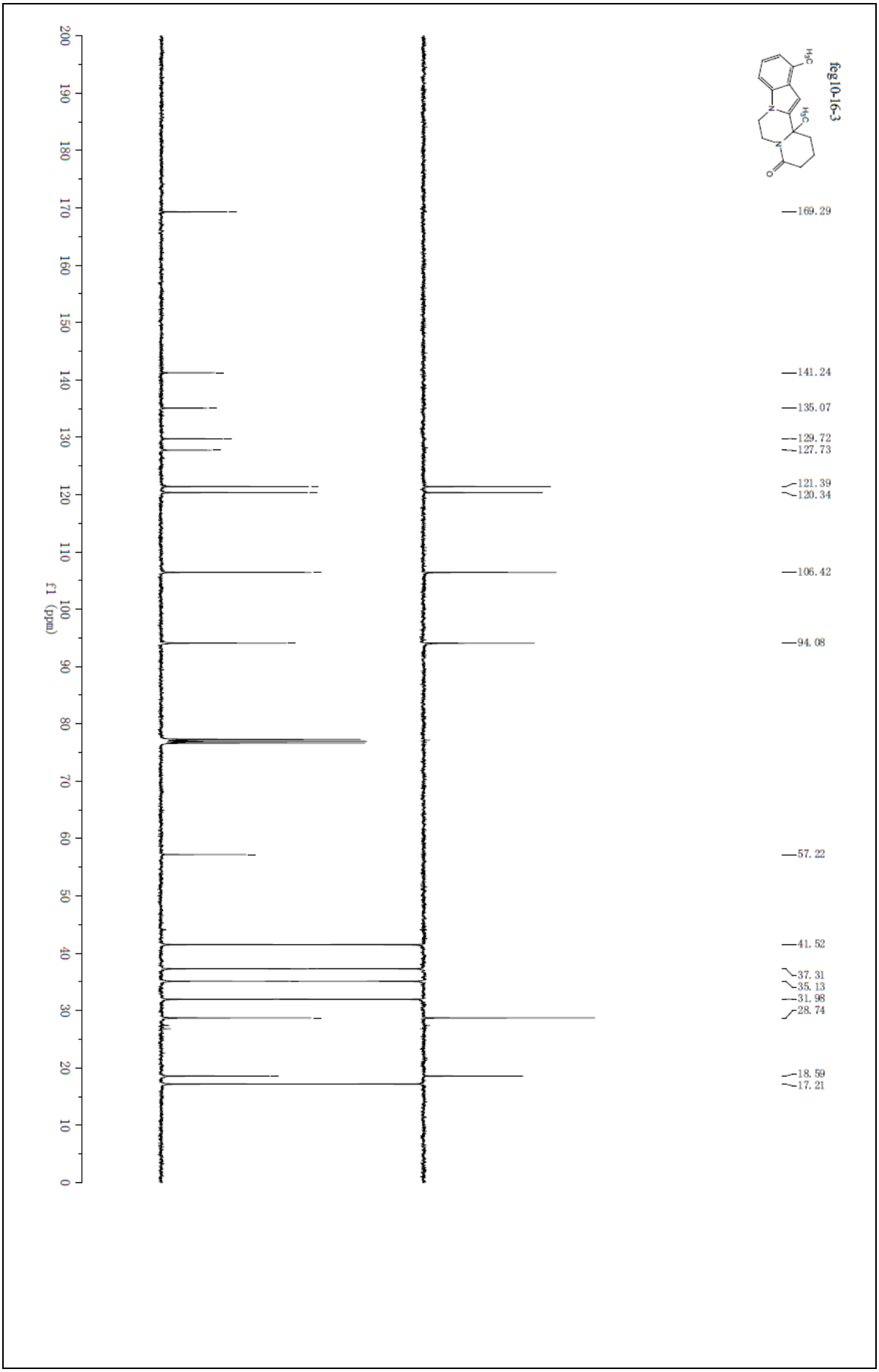
Compound 3Aa



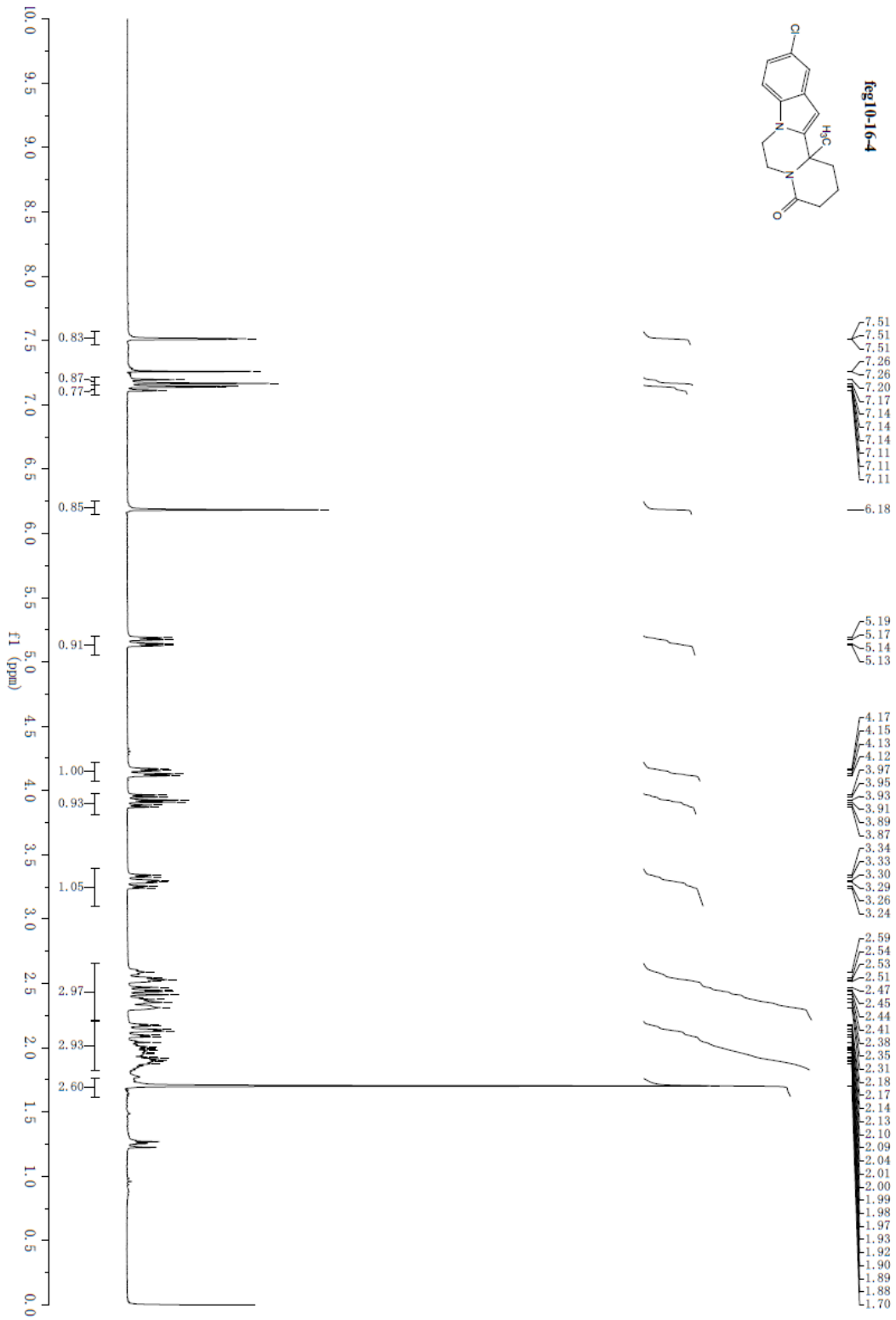


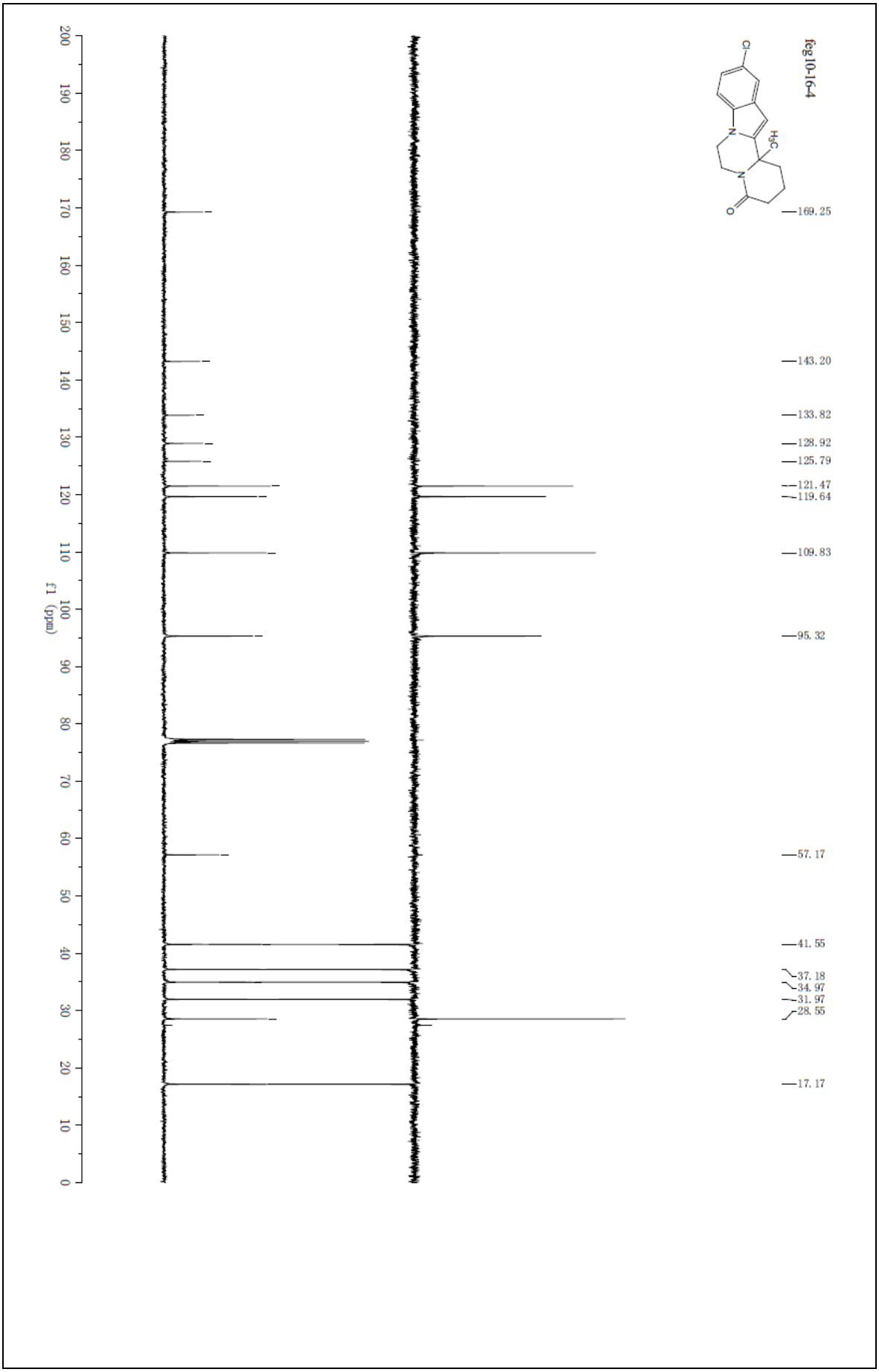
Compound 3Ab



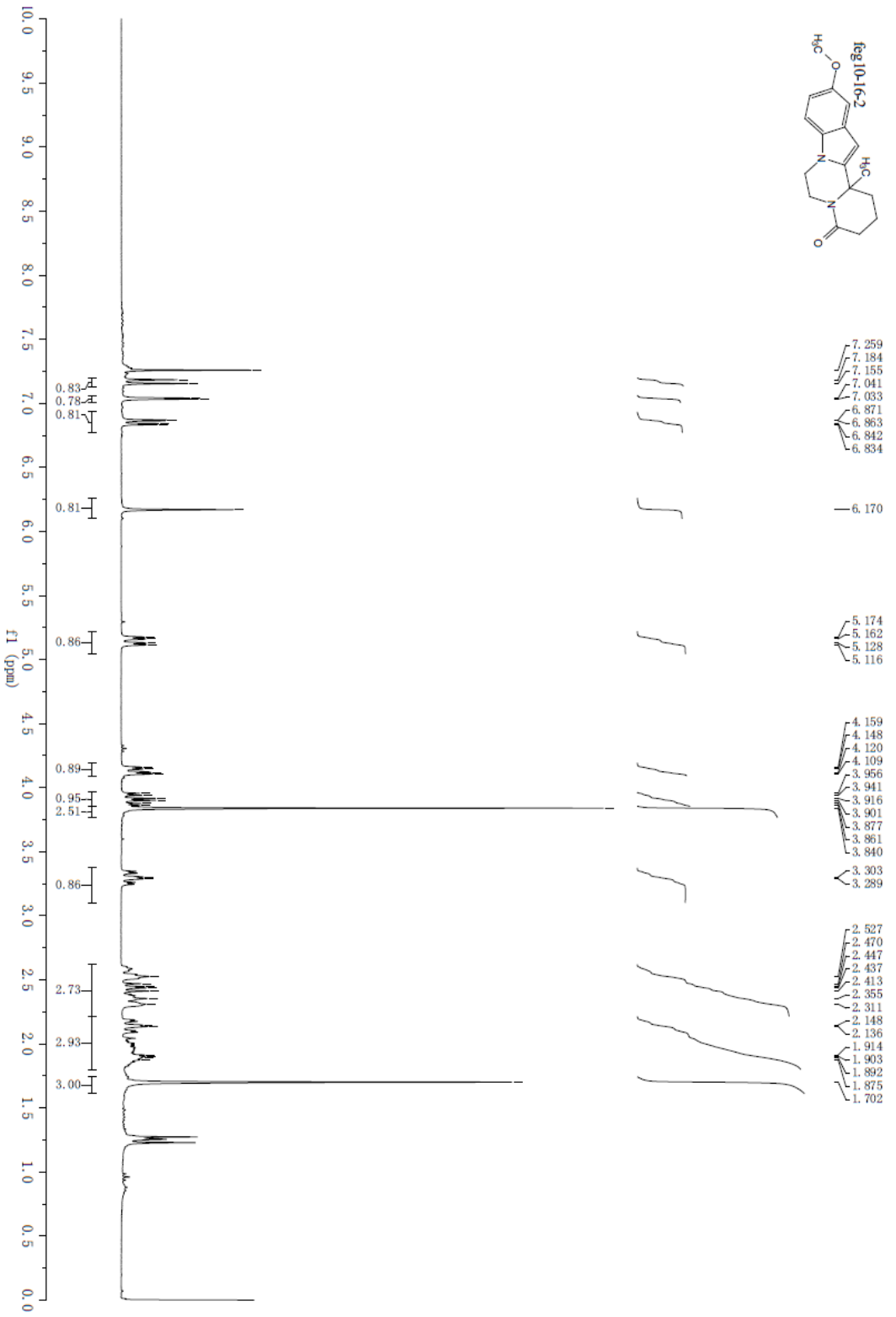

feg10-16-3
169.29
141.24
135.07
129.72
127.73
121.39
120.34
106.42
94.08
57.22
41.52
37.31
35.13
31.98
28.74
18.59
17.21
f1 (ppm)

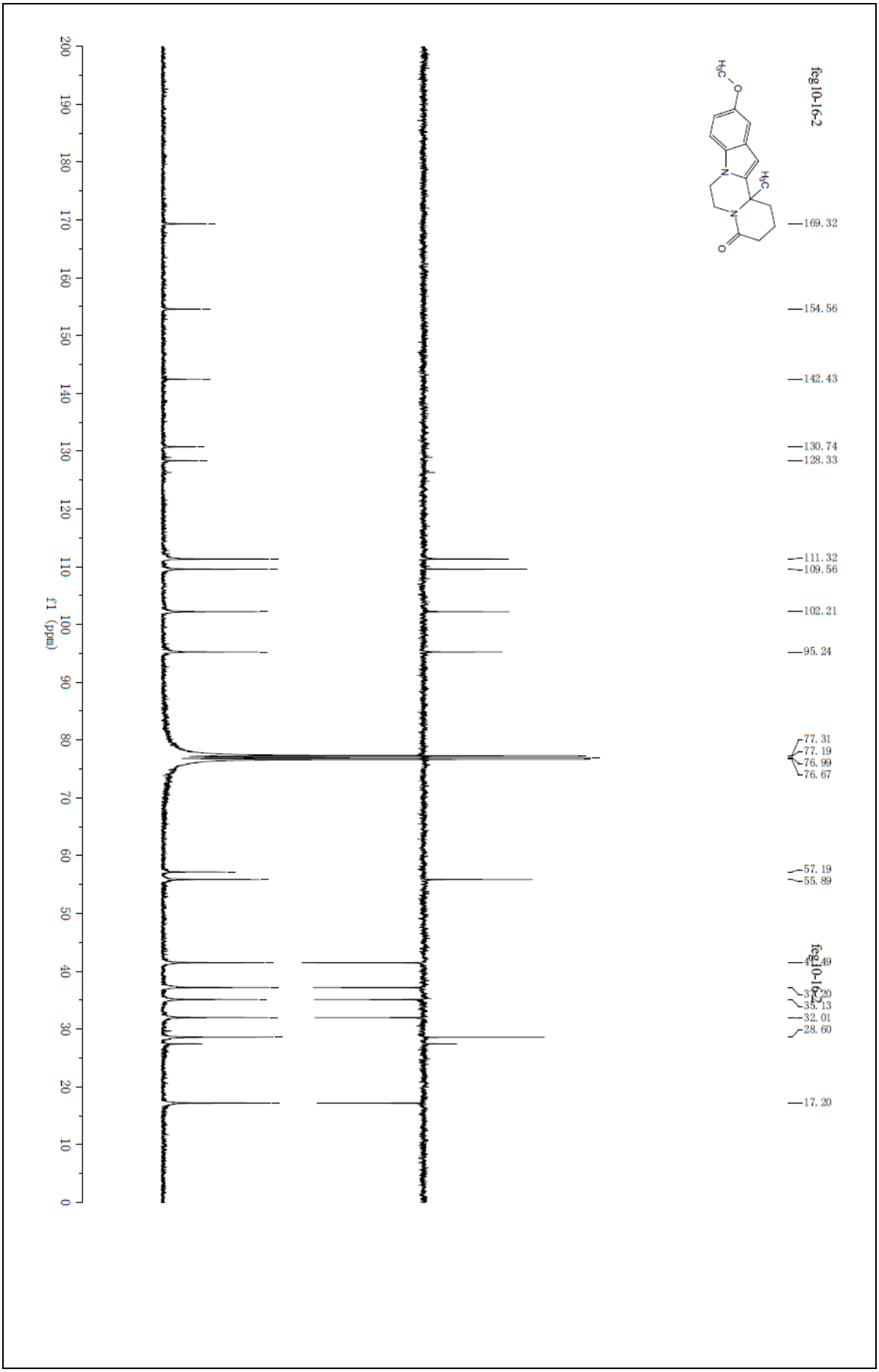
Compound 3Ac



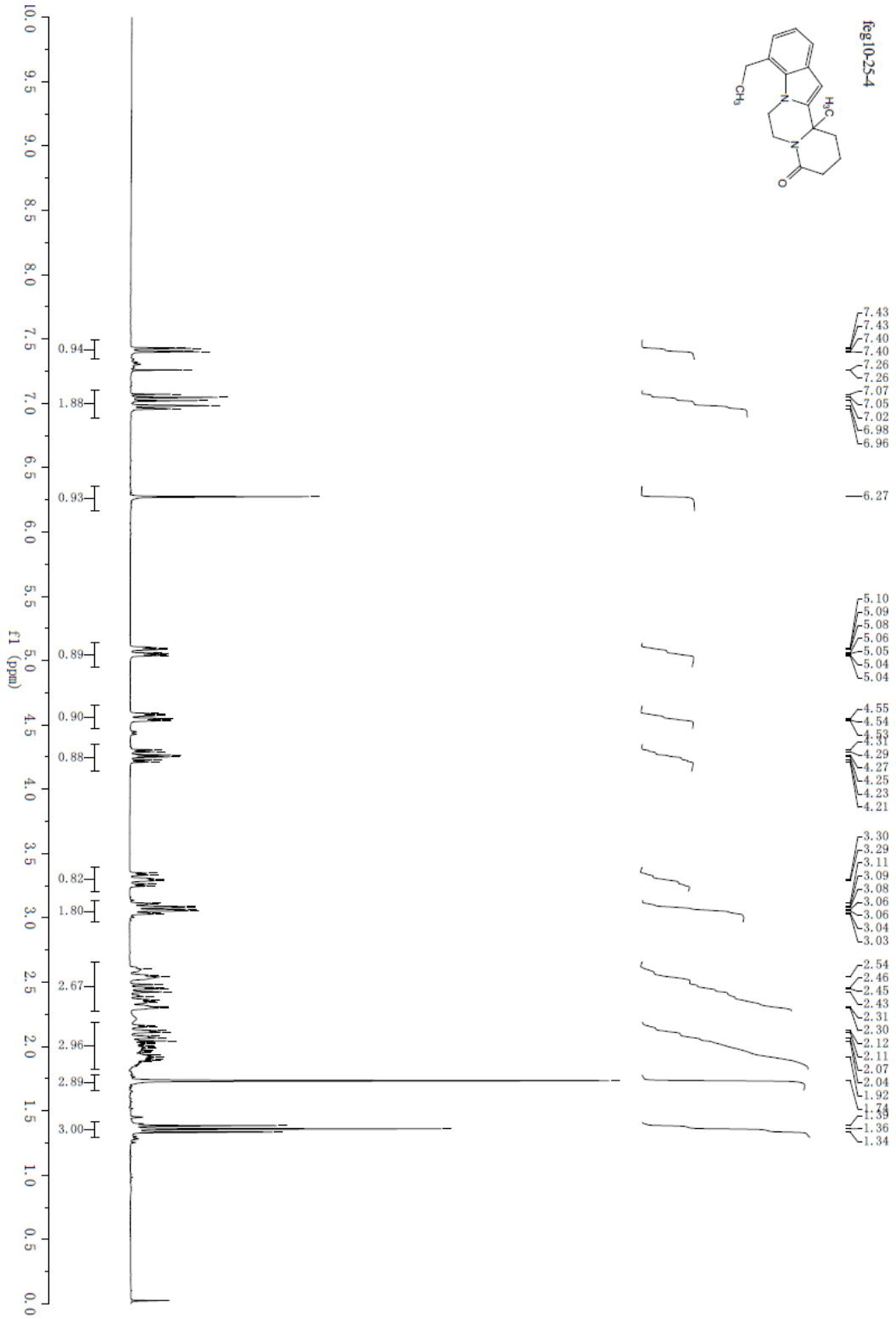

feg10-16-4
Cl
N
H3C
N
O
169.25
143.20
133.82
128.92
125.79
121.47
119.64
109.83
95.32
57.17
41.55
37.18
34.97
31.97
28.55
17.17
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

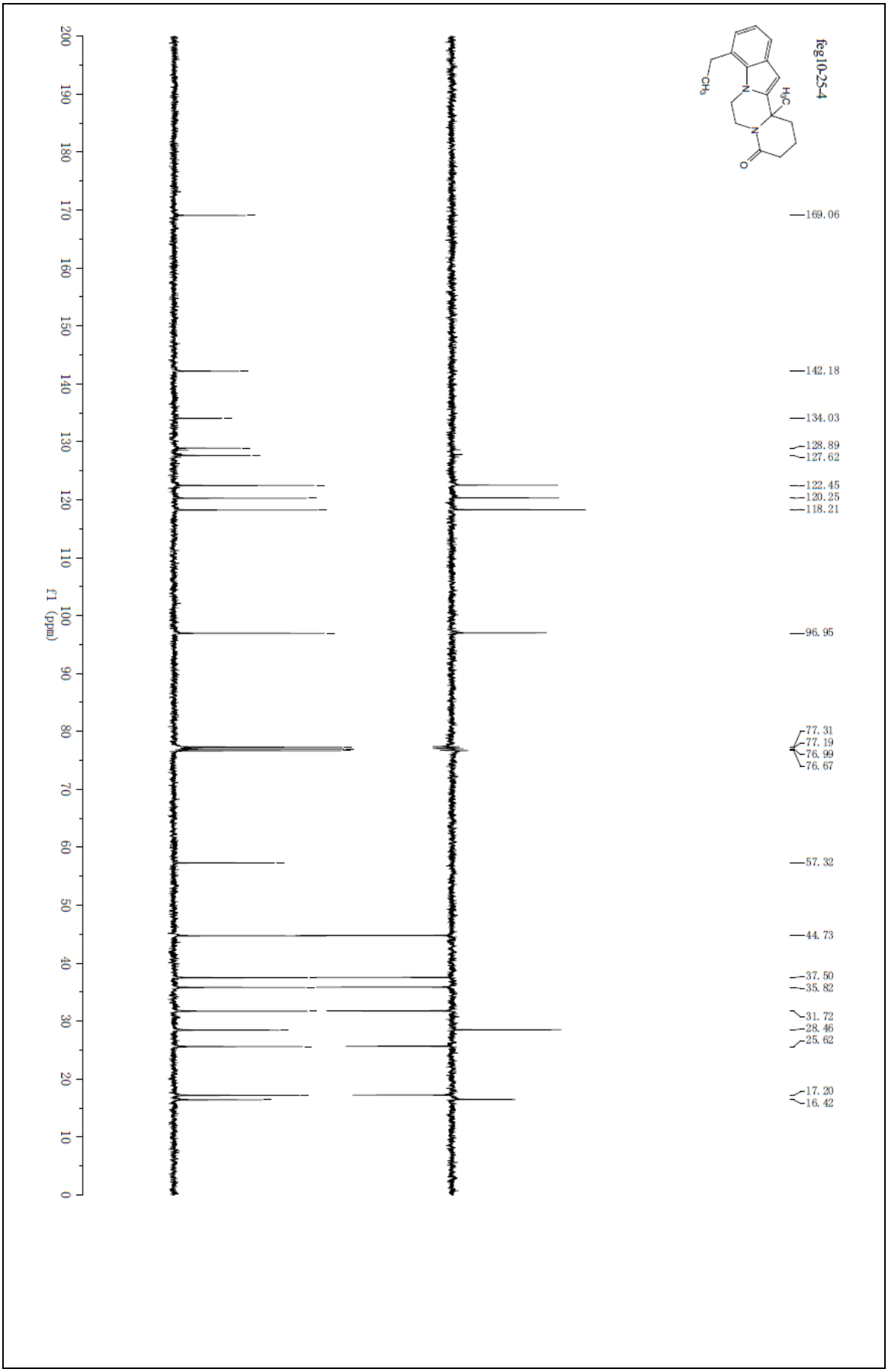
Compound 3Ad



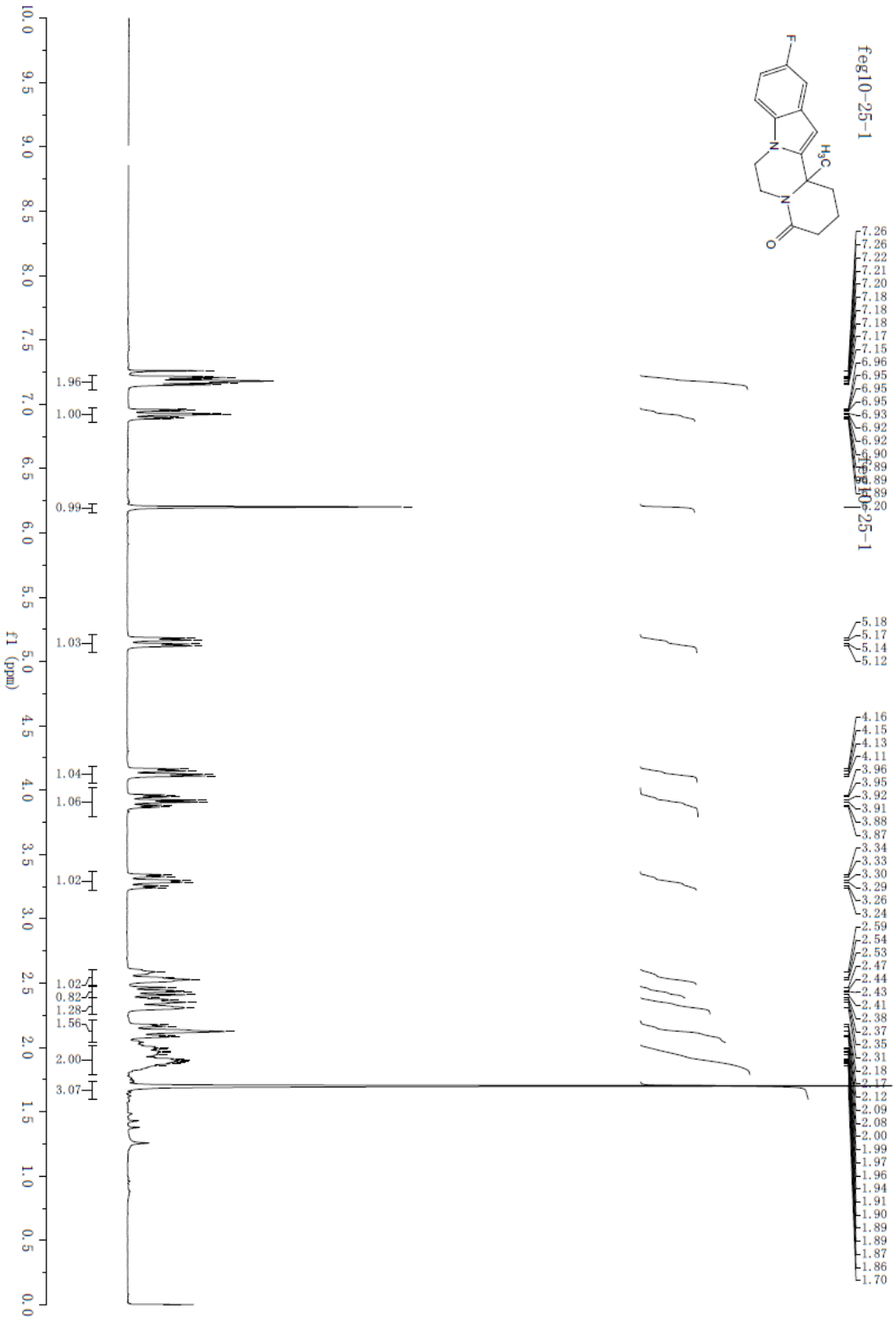


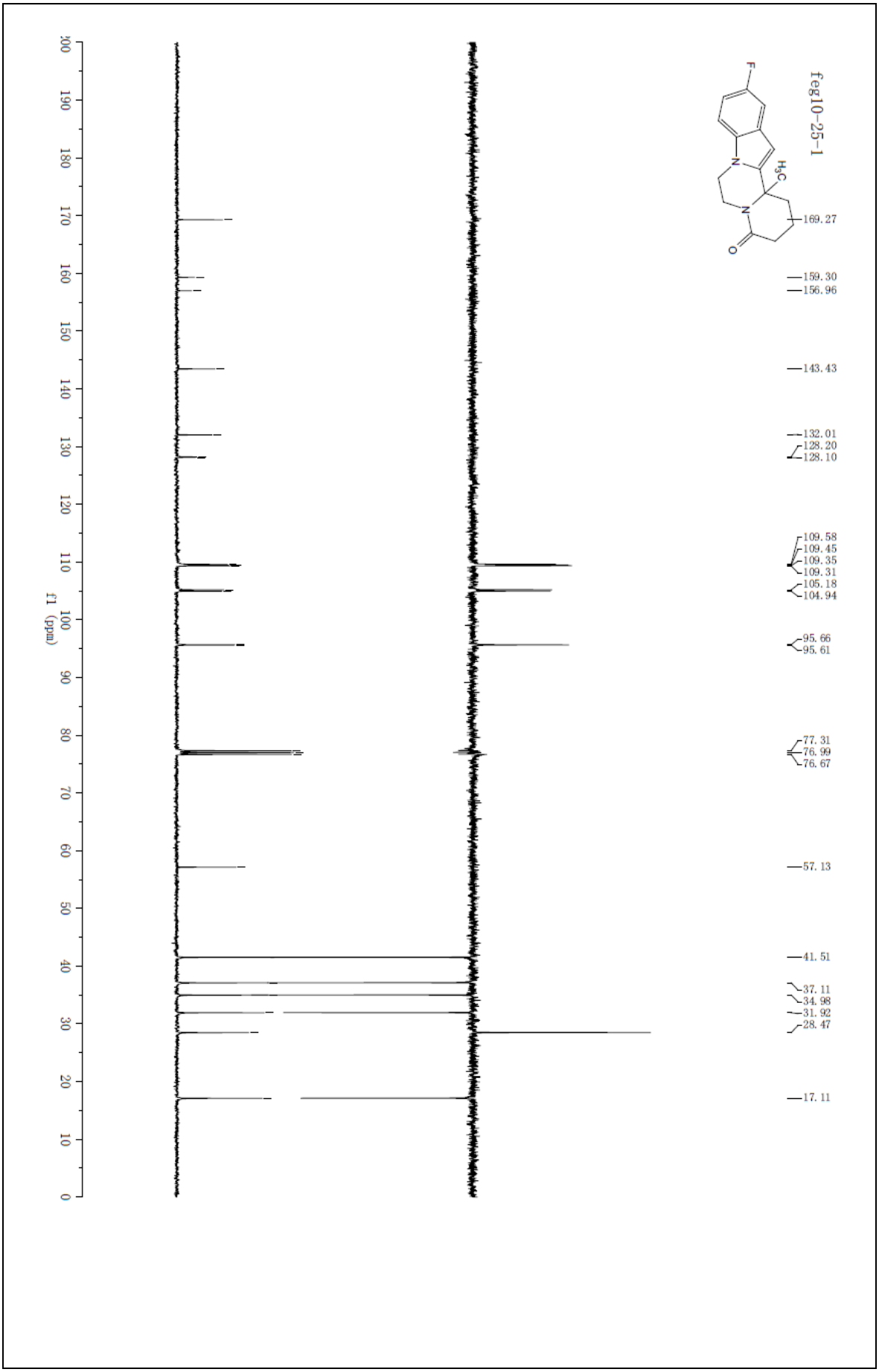
Compound 3Ae



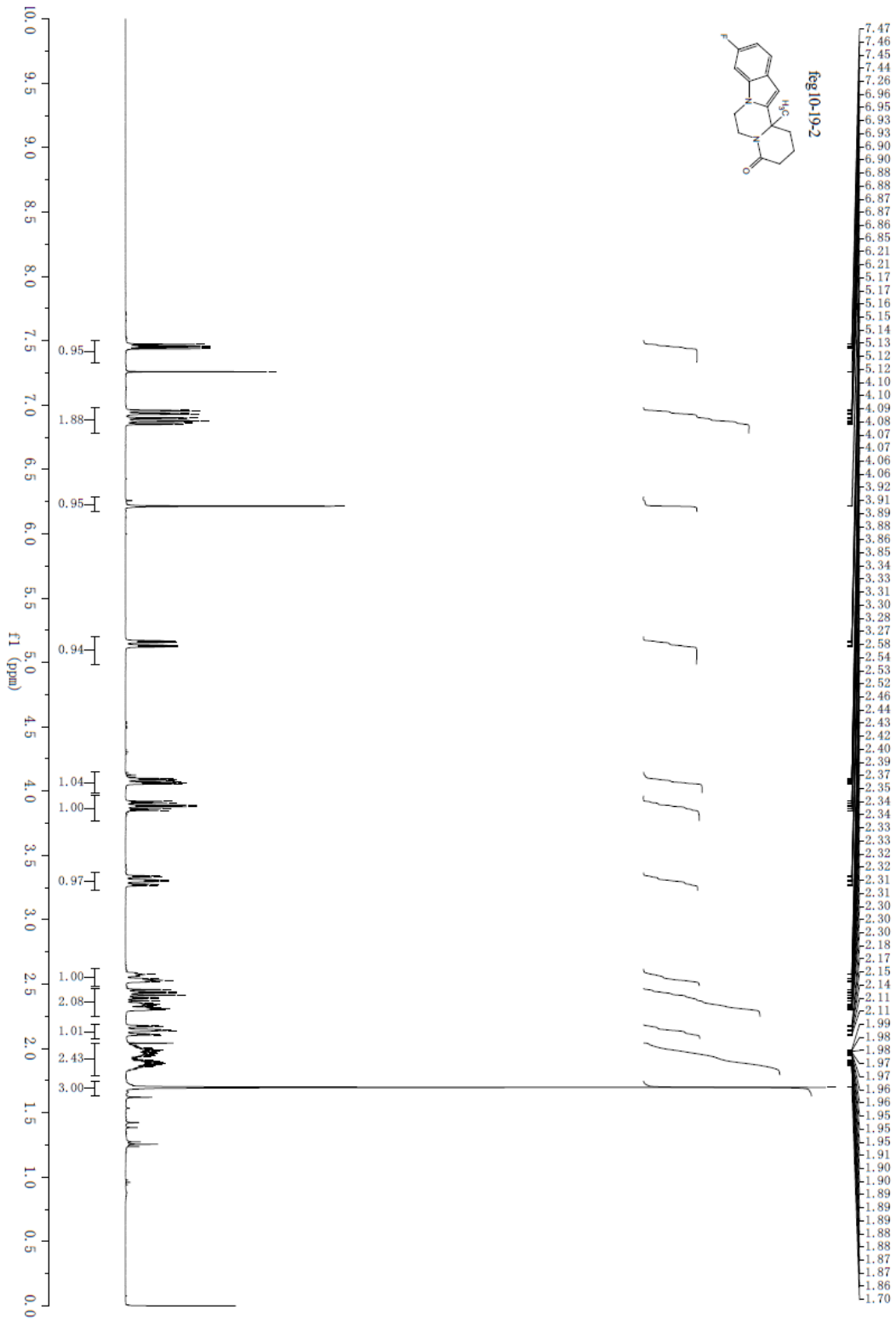

fgg10-254
169.06
142.18
134.03
128.89
127.62
122.45
120.25
118.21
96.95
77.31
77.19
76.99
76.67
57.32
44.73
37.50
35.82
31.72
28.46
25.62
17.20
16.42
f1 (ppm)

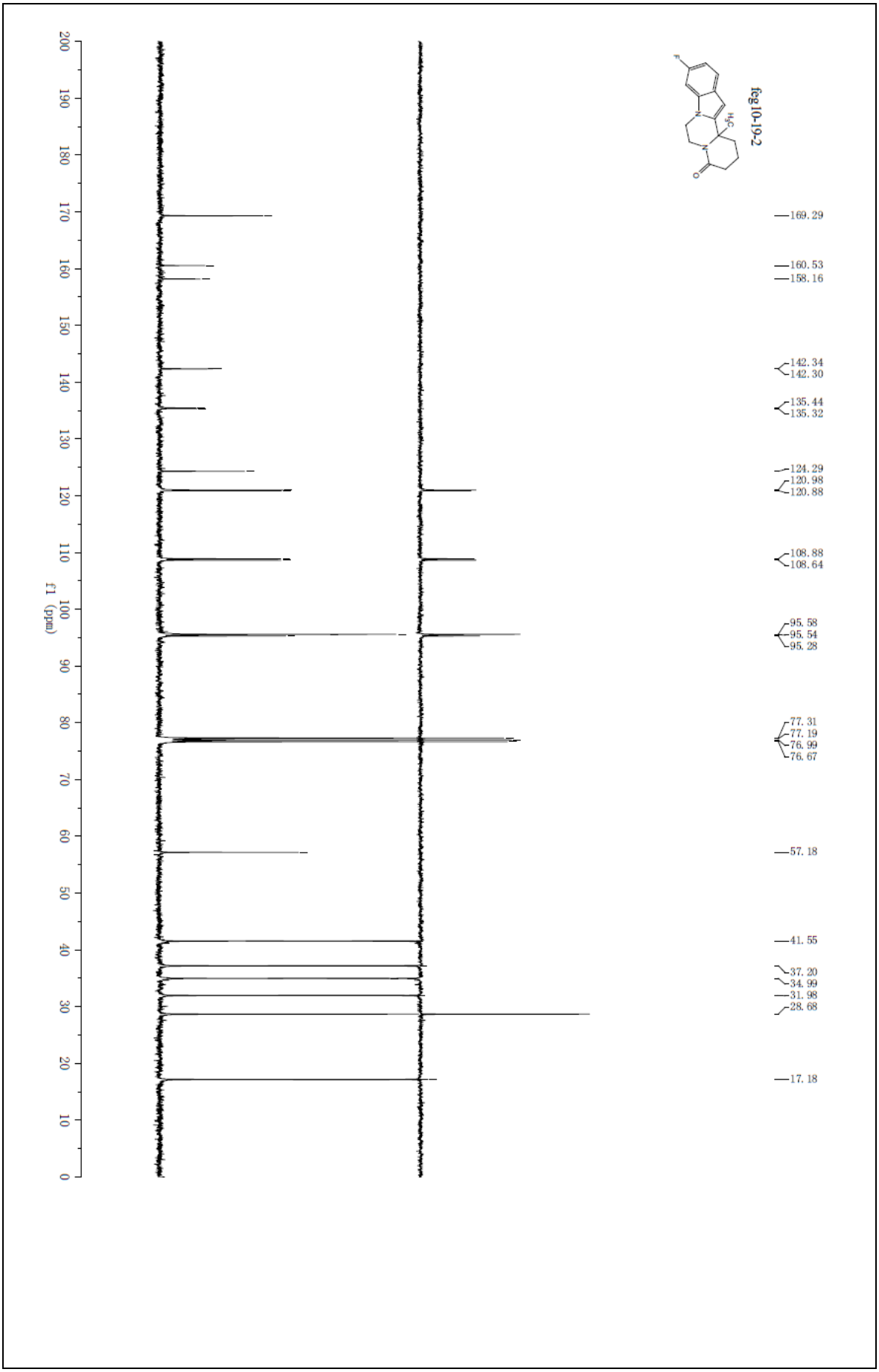
Compound 3Af



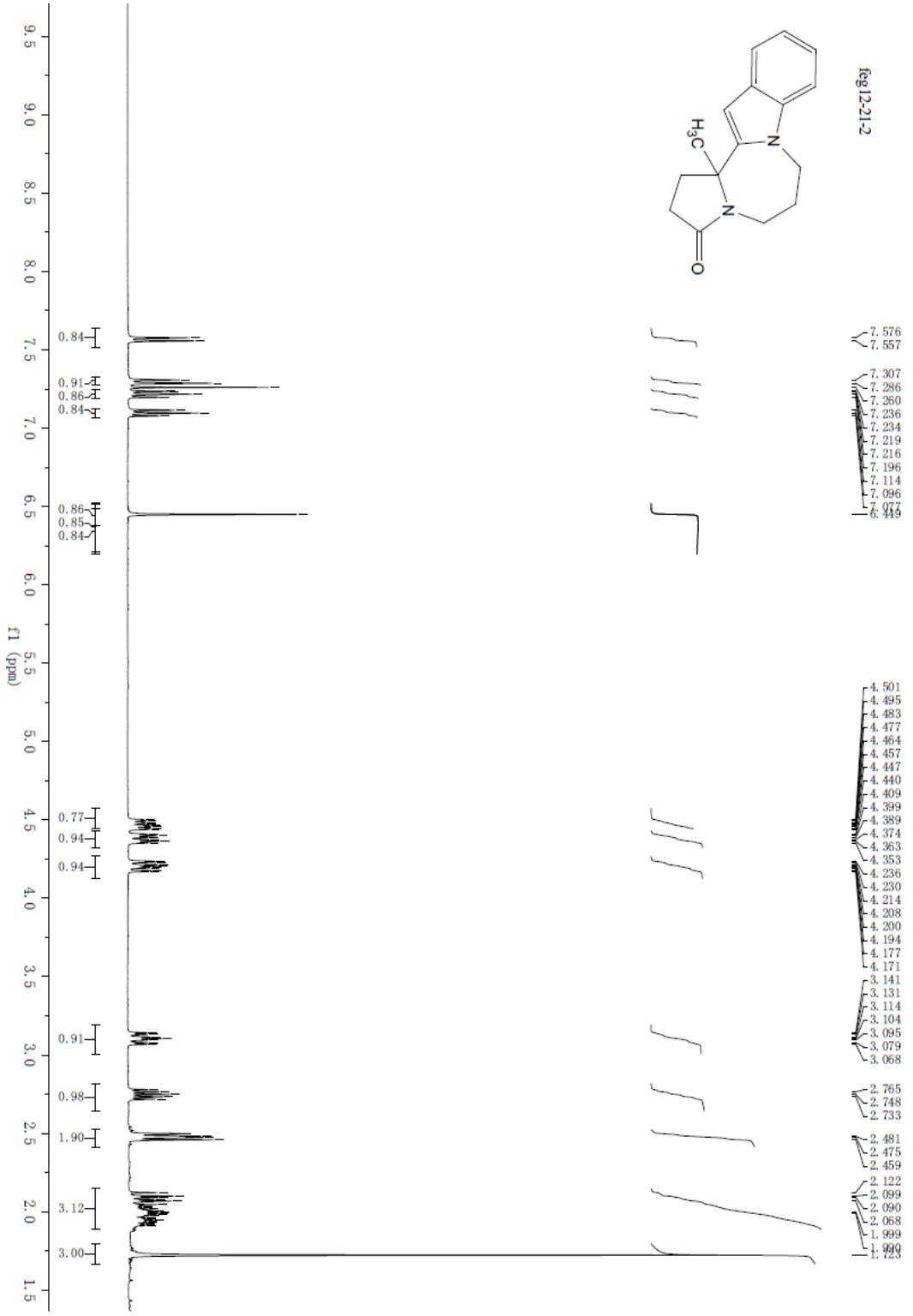

feg10-25-1
F
H3C
N
N
O
169.27
159.30
156.96
143.43
132.01
128.20
128.10
109.58
109.45
109.35
109.31
105.18
104.94
95.66
95.61
77.31
76.99
76.67
57.13
41.51
37.11
34.98
31.92
28.47
17.11
f1 (ppm)

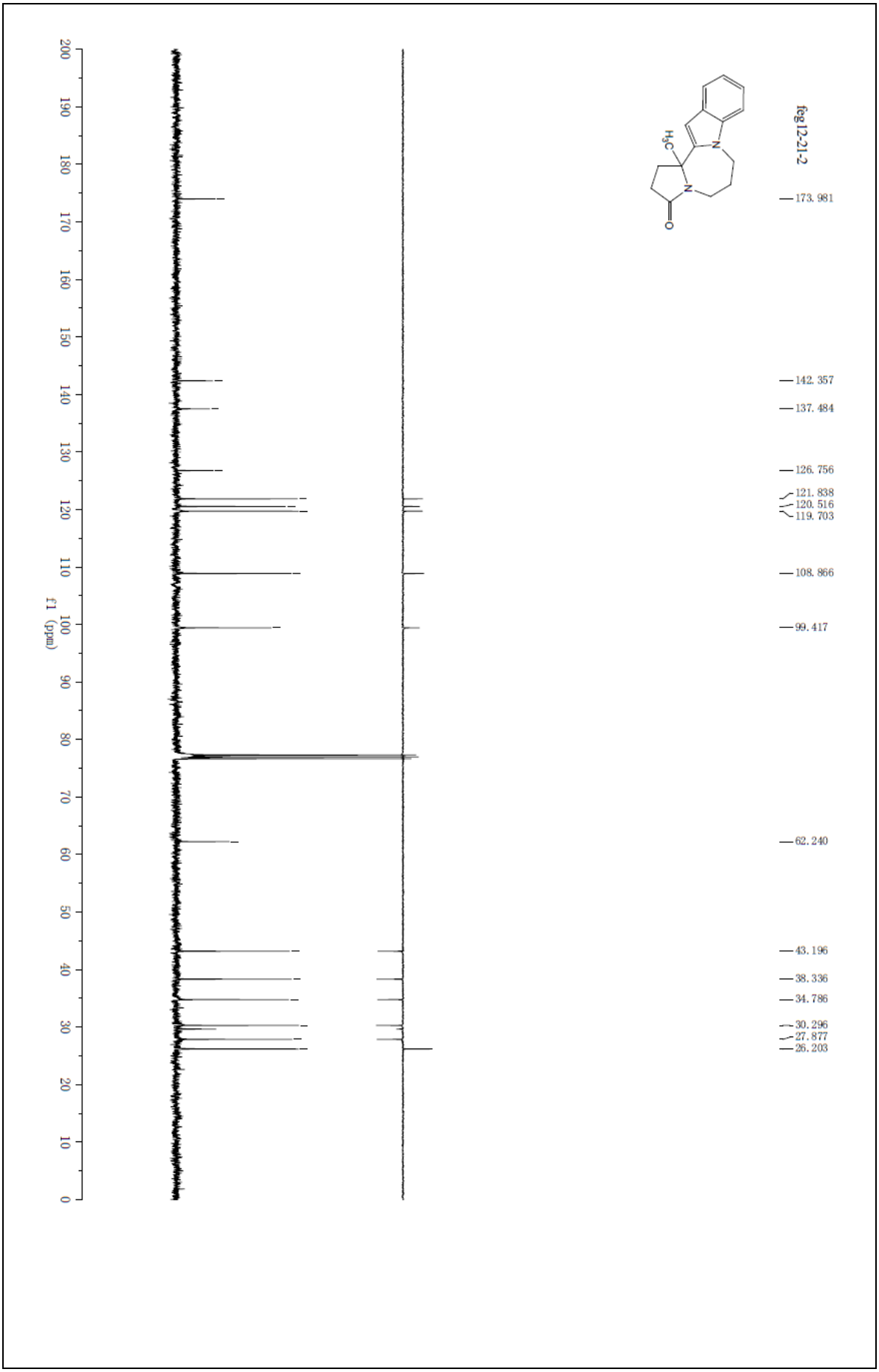
Compound 3Ag



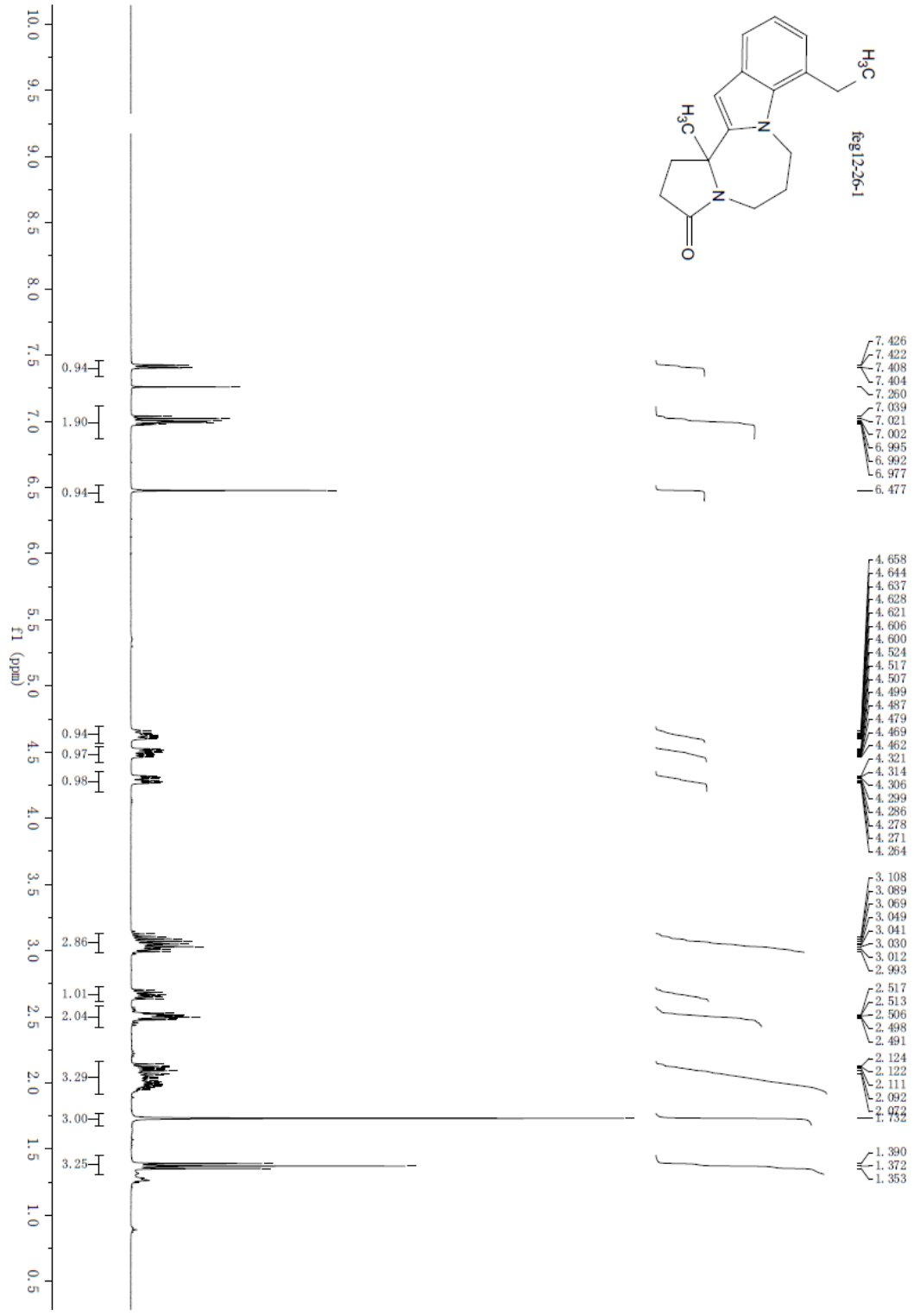

fcg10-19-2
169.29
160.53
158.16
142.34
142.30
135.44
135.32
124.29
120.98
120.88
108.88
108.64
95.58
95.54
95.28
77.31
77.19
76.99
76.67
57.18
41.55
37.20
34.99
31.98
28.68
17.18
f1 (ppm)

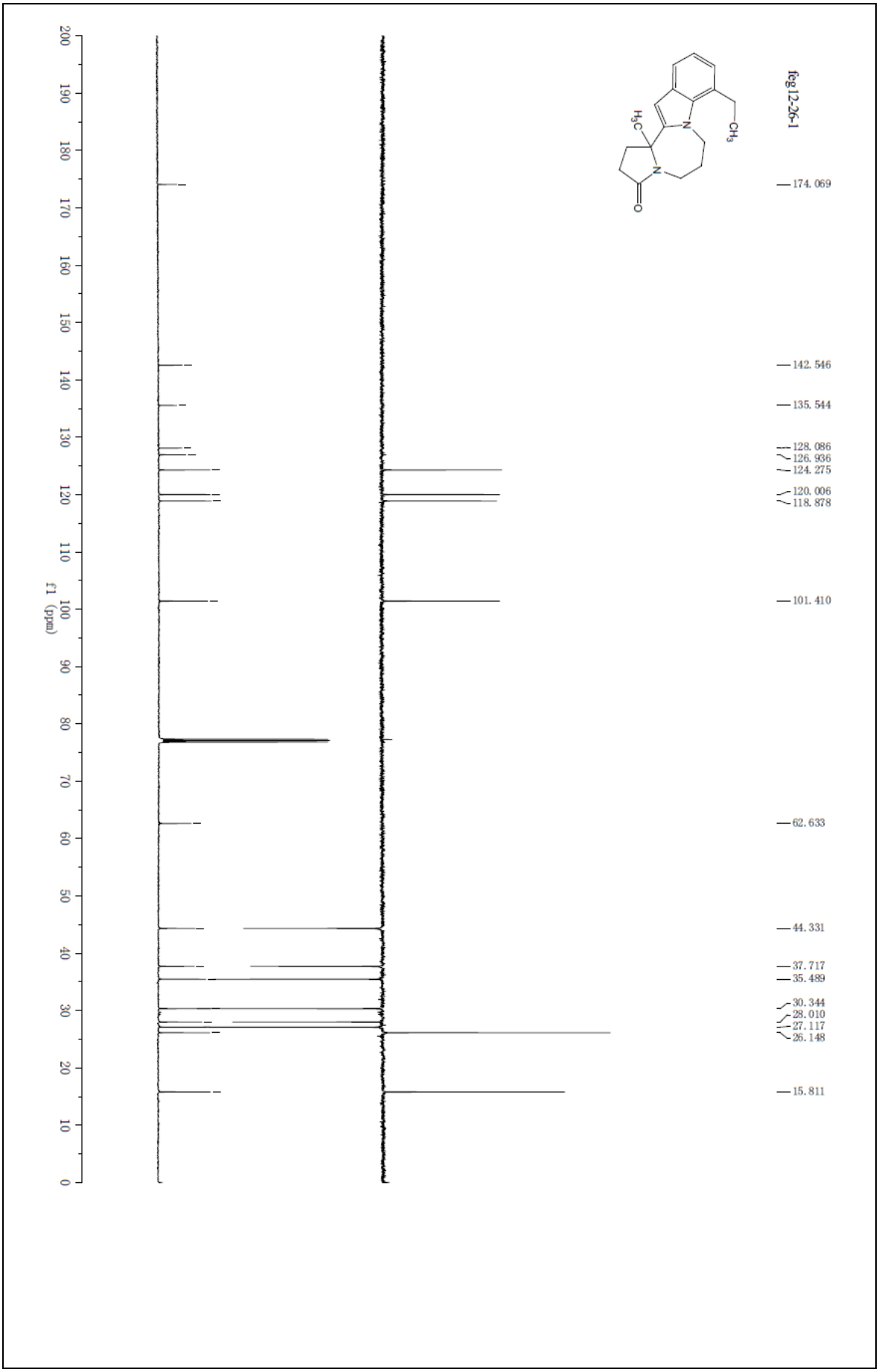
Compound 3Ah



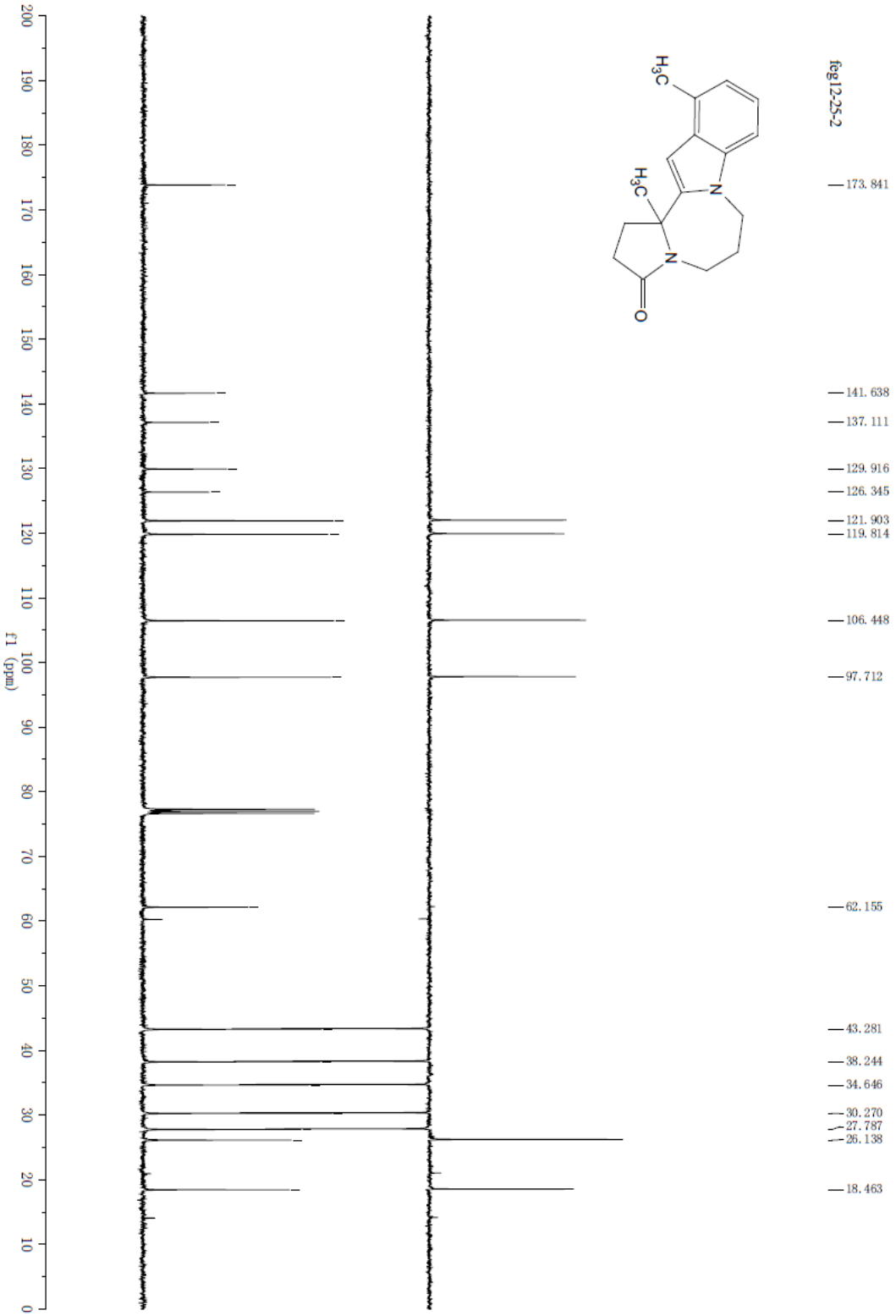

feg12-21-2
173.981
142.357
137.484
126.756
121.838
120.516
119.703
108.866
99.417
62.240
43.196
38.336
34.786
30.296
27.877
26.203
f1 (ppm)

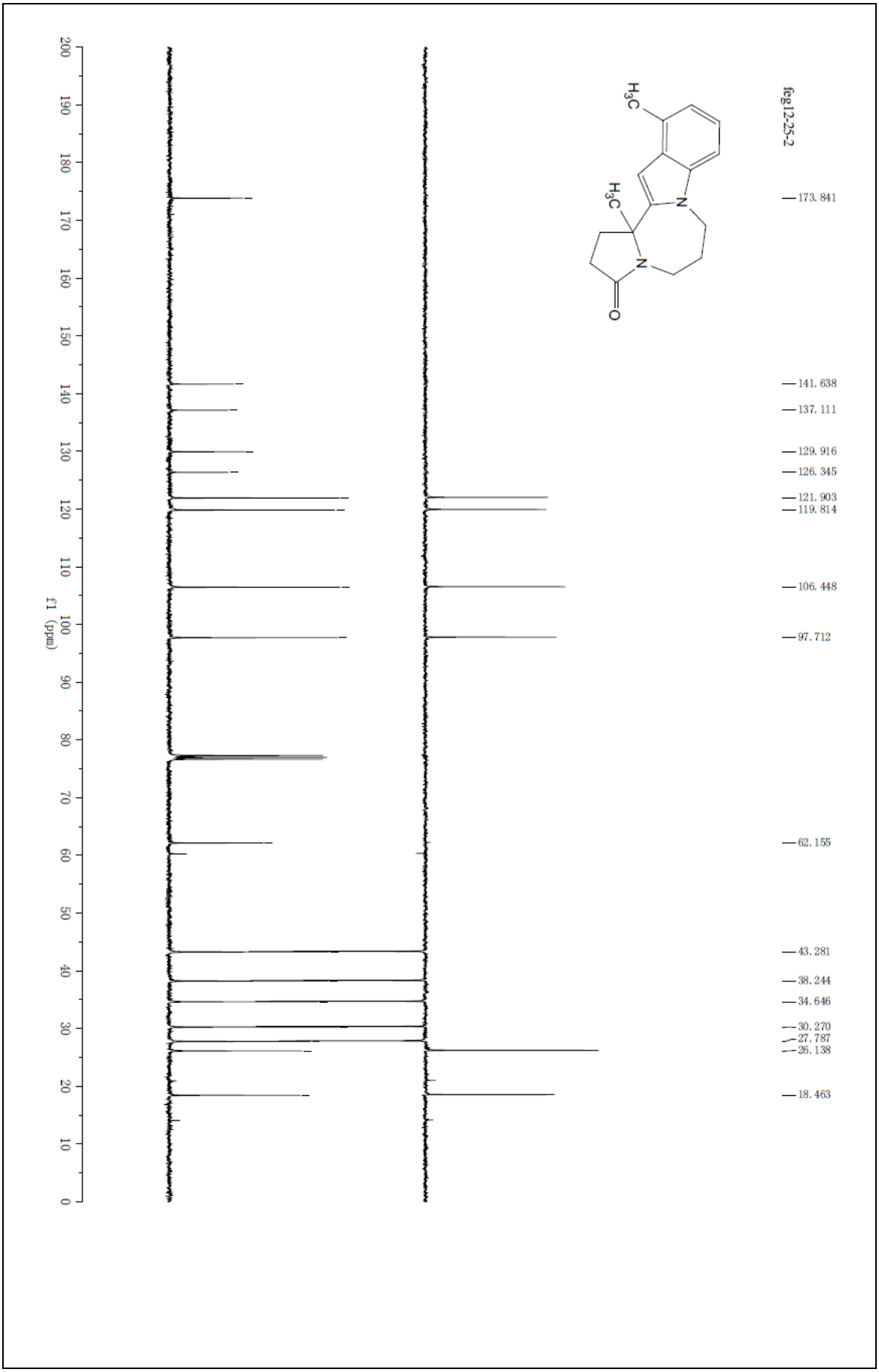
Compound 3Ai



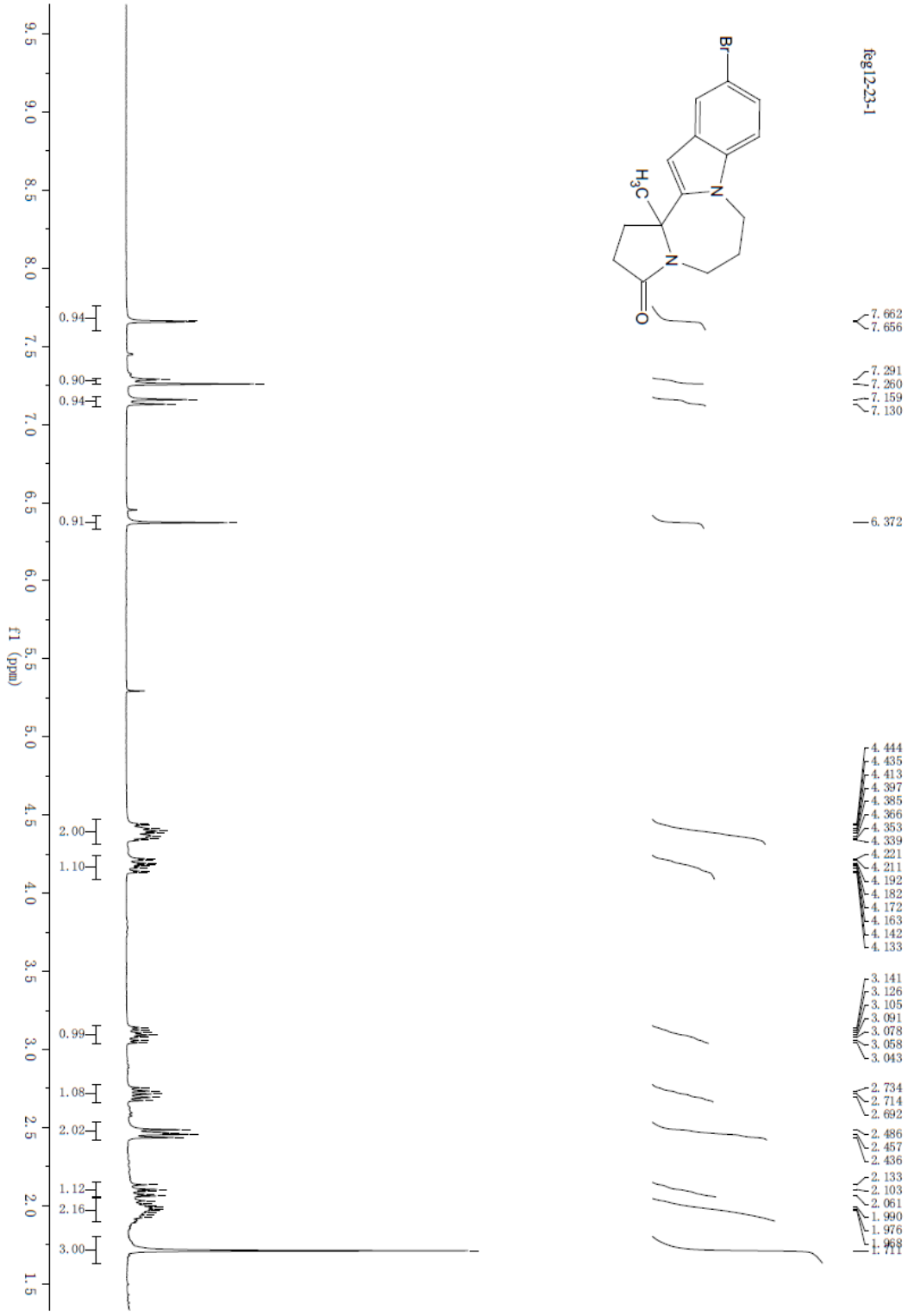

feg12-26-1
174.069
142.546
135.544
128.086
126.936
124.275
120.006
118.878
101.410
62.633
44.331
37.717
35.489
30.344
28.010
27.117
26.148
15.811
f1 (ppm)

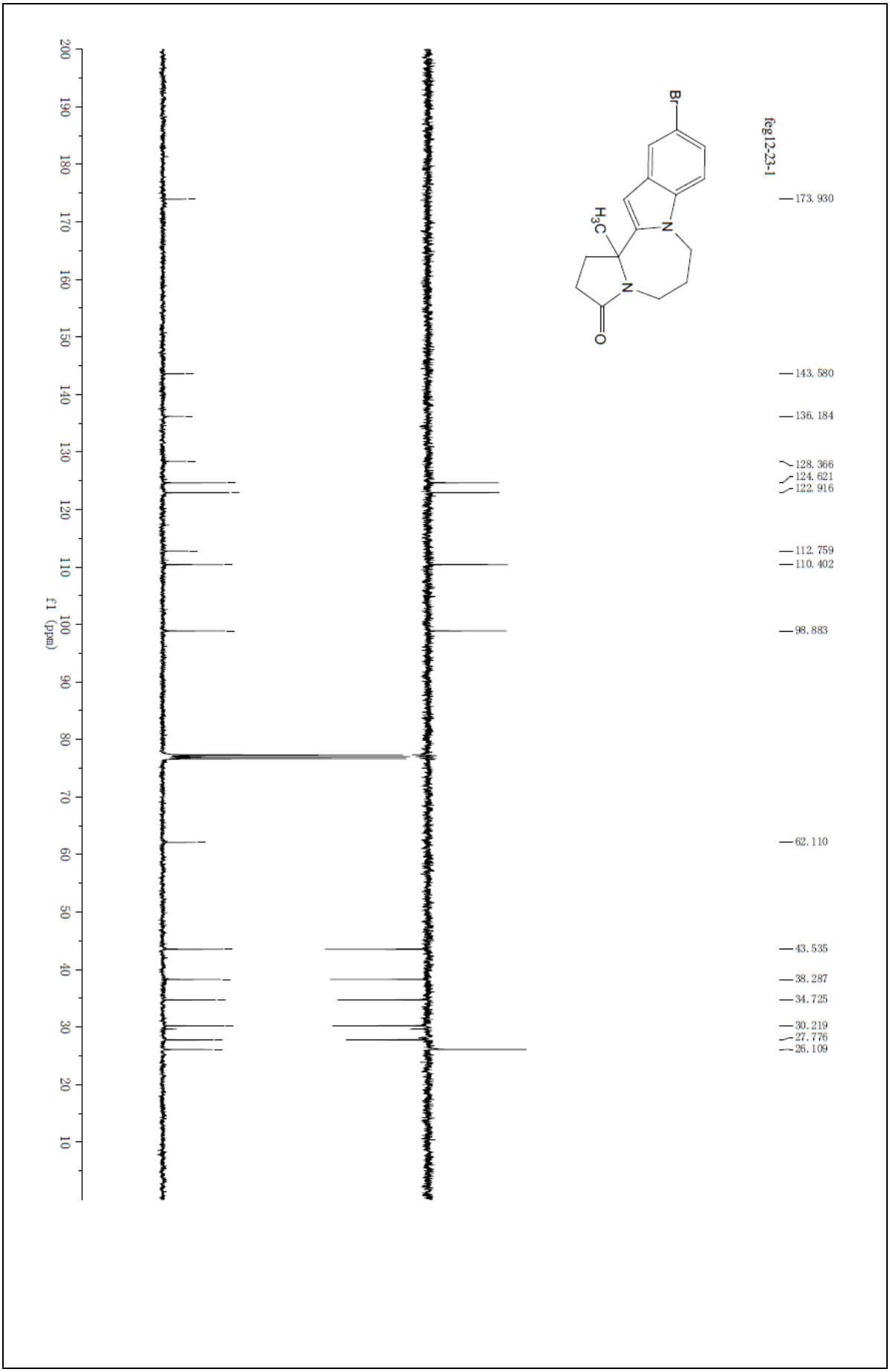
Compound 3Aj



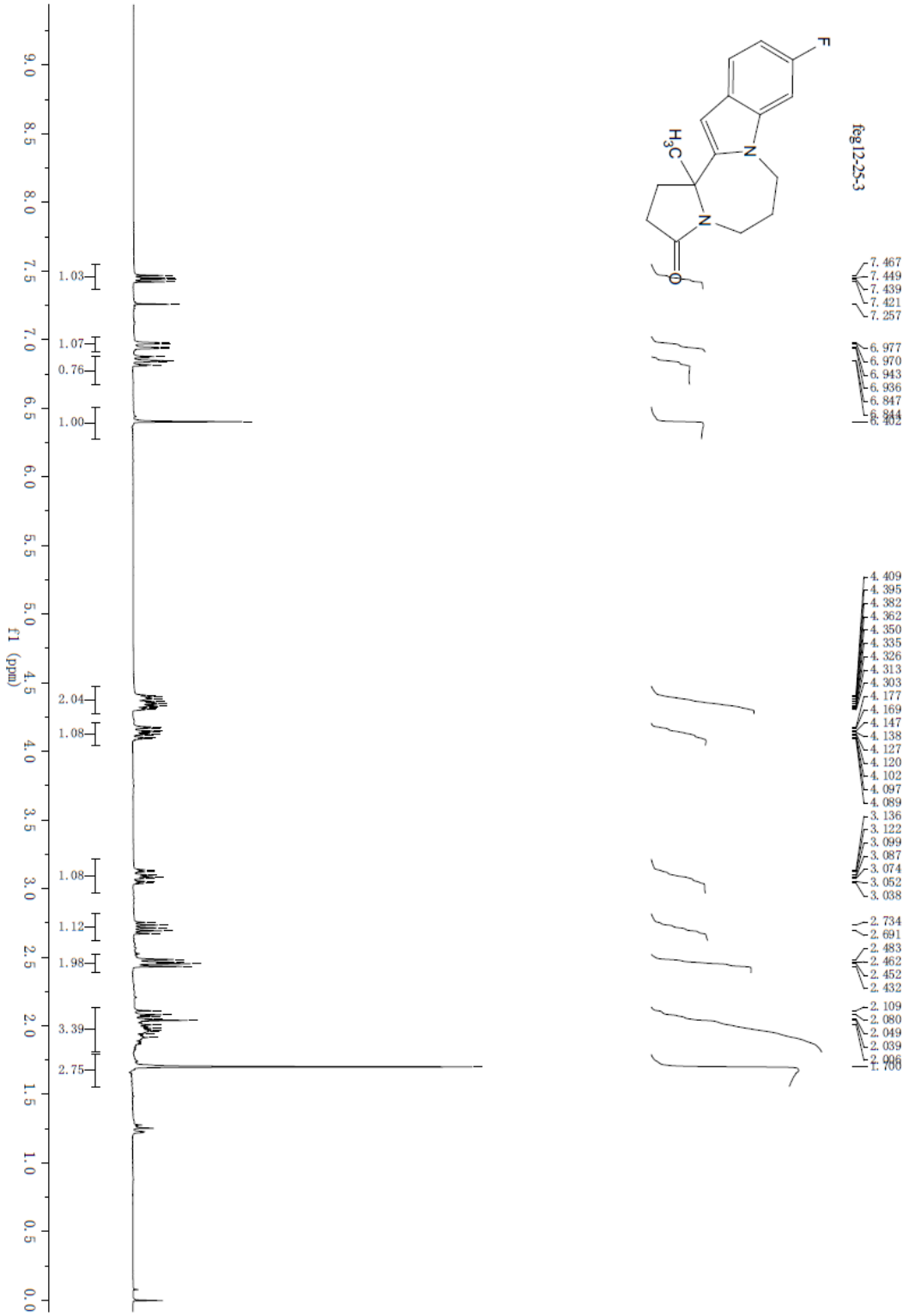


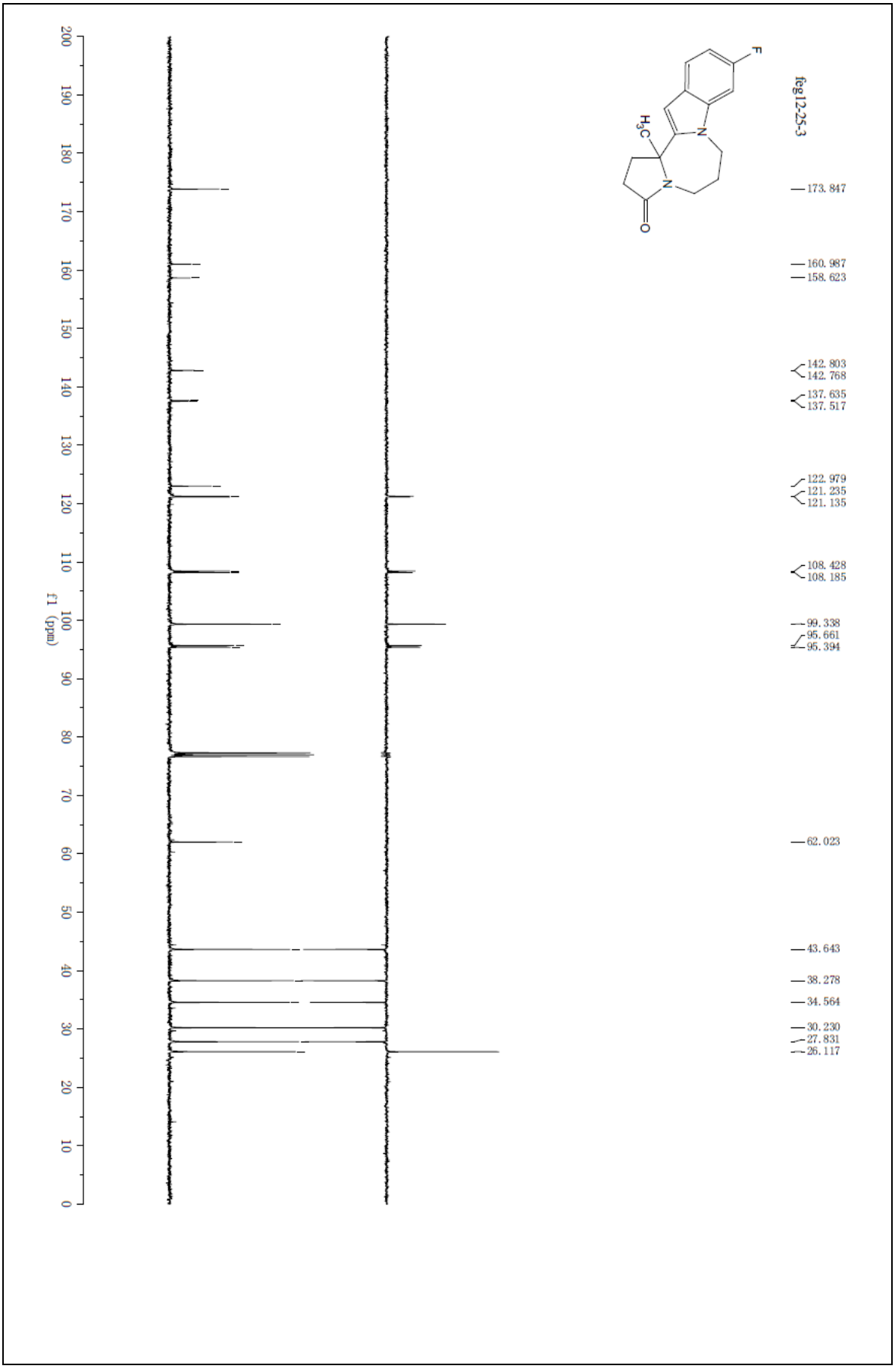
Compound 3Ak





Compound 3Al




feg12-25-3
F
H3C
N
N
O
173.847
160.987
158.623
142.803
142.768
137.635
137.517
122.979
121.235
121.135
108.428
108.185
99.338
95.661
95.394
62.023
43.643
38.278
34.564
30.230
27.831
26.117
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0